

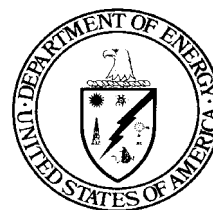
Appendix B

Pilot Test Summary Reports

- Building 71B Bioremediation Pilot Test (March 2004)
- Building 71B In-Situ Chemical Oxidation Pilot Test (May 2004)
- Building 51L In-Situ Chemical Oxidation Pilot Test (August 2004)



E.O. Lawrence Berkeley National Laboratory
University of California
Environmental Restoration Program



United States Department of Energy

BUILDING 71B BIOREMEDIATION PILOT TEST

for the
Lawrence Berkeley National Laboratory
Environmental Restoration Program

March 2004

BUILDING 71B BIOREMEDIATION PILOT TEST

for the
Lawrence Berkeley National Laboratory
Environmental Restoration Program

*A Joint Effort of
Environment, Health and Safety Division and
Earth Sciences Division*
Lawrence Berkeley National Laboratory
Berkeley, CA 94720

March 2004

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This work was done at the Lawrence Berkeley National Laboratory, which is operated by the University of California for the U. S. Department of Energy under contract DE-AC03-76SF00098.

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SECTION 1

INTRODUCTION

The Ernest Orlando Lawrence Berkeley National Laboratory (Berkeley Lab) is currently in the Corrective Measures Study (CMS) phase of the Resource Conservation and Recovery Act (RCRA) Corrective Actions Process (CAP). The Berkeley Lab Environmental Restoration Program (ERP) implements the CAP at Berkeley Lab.

As part of the CMS process, Berkeley Lab proposed in consultation with the regulatory agencies, various pilot-scale tests designed to evaluate different remedial technologies. Pilot-scale testing involves operation of potential remedial technologies on a small-scale to assess their applicability and potential effectiveness under site-specific Berkeley Lab conditions. Pilot test results can be used to optimize the design and operation of the full-scale corrective measure, should it be implemented.

A work plan titled “Work Plan for Pilot Testing Hydrogen Release Compounds (HRC[®]) in the Core Area of the Building 71b Lobe of the Building 71 Groundwater Solvent Plume” was prepared describing the proposed pilot-scale test. The work plan describes the rationale and procedures for injecting HRC[®] into the core area of the Building 71B lobe of the Building 71 groundwater contaminant plume (Figure 1) to degrade chlorinated aliphatic hydrocarbons (CAHs), including tetrachloroethene (PCE), trichloroethene (TCE), dichloroethene (DCE) and vinyl chloride (VC), using enhanced *in situ* bioremediation techniques. Addition of HRC[®] enhances natural bioremediation by supplying electron acceptors that accelerate the metabolic activity of indigenous microorganisms that transform or destroy contaminants.

Most organisms obtain energy for growth and activity by physiologically coupling oxidation and reduction reactions and harvesting the resulting chemical energy. When molecular oxygen is available (aerobic conditions), many organisms including humans couple the oxidation of the organic compounds (primary growth substrate or food) to the

reduction of oxygen. (Oxygen is the electron acceptor and is reduced and the organic compound is the electron donor and is oxidized). Many microorganisms can still oxidize organic compounds when oxygen is absent (anaerobic conditions) by using other electron acceptors including nitrate (NO_3^-), manganese (Mn^{4+}), ferric iron (Fe^{3+}), sulfate (SO_4^-), and carbon dioxide (CO_2). The oxidation process, however, extracts smaller amounts of energy, and is therefore less effective, as the oxidation-reduction reactions progress from nitrate to carbon dioxide. Microorganisms use petroleum hydrocarbons and some chlorinated aliphatic hydrocarbons (CAHs), like vinyl chloride, as the primary growth substrate.

In comparison, very little evidence is available suggesting that the highly chlorinated CAHs including tetrachloroethene (PCE), trichloroethene (TCE), DCEs, trichloroethenes (TCAs), dichloroethanes (DCAs), and polychlorinated benzenes undergo biodegradation by either aerobic or anaerobic oxidation. This is due to the fact that these compounds are already highly oxidized. In addition, these compounds are not amenable for use as a primary growth substrate because they may be toxic to the bacteria. Instead, the CAHs are used as electron acceptors in reactions that rely on other sources of carbon as the primary growth substrate. Other sources of carbon can include low molecular weight organic compounds (e.g., lactate, acetate, methanol, glucose, etc.), petroleum hydrocarbons, volatile fatty acids (e.g., lactic, acetic, pyruvic, and butyric acid) or naturally occurring organic matter.

Biodegradation of CAHs generally occurs under reducing (i.e., anaerobic) conditions and is referred to as reductive dechlorination. During this process, the CAH is reduced and a chlorine atom is removed and replaced with a hydrogen atom. In general, the success of reductive dechlorination is limited to the existence of a reducing environment, the availability of primary growth substrate and hydrogen, both of which may be consumed by other bacteria competing for these constituents, and the presence of the microorganisms that degrade these compounds.

HRC[®] is formulated by the manufacturer to serve as both a source of carbon and hydrogen, thus enhancing redox conditions and microbial populations that favor bioremediation. HRC[®] contains a sugar base that acts as a growth substrate for the

microorganisms and, when hydrated in water, slowly releases lactic acid. Lactic acid degrades to lower molecular weight volatile fatty acids (i.e., pyruvic and acetic acid), producing hydrogen that the bacteria substitute in the CAH structure for chlorine, yielding energy for their metabolism and a biodegraded CAH. If full dechlorination of the CAH occurs, then the end product is typically ethene, ethane, methane, carbon dioxide and/or water.

The sections that follow describe the results of the pilot-scale study.

SECTION 2

PUMPING WELL TEST

A 96-hour long pumping well test was performed at Building 71B to characterize the water-bearing zone penetrated by the pumping well MW71b-98-13 (Figure 2) and to determine the feasibility of injecting HRC[®] into the zone. The pumping well test was performed from August 7 to August 11, 2003, prior to injecting HRC[®] on September 26, 2003 (Section III).

During the test, monitoring wells SB71b-99-1, SB71b-99-2 and MW71B-00-2 were utilized as observation wells for measuring water level changes caused by pumping. Water pumped from MW71b-98-13 was also sampled and analyzed for wellhead parameters (dissolved oxygen, specific conductance and temperature) and volatile organic compounds (VOCs by EPA method 8260). A complete description of the pumping well test and results can be found in Appendix A.

Data collected during the pumping well test were evaluated and interpreted as follows:

- The water levels and pumping rate did not stabilize during the test making it difficult to estimate formation properties (hydraulic conductivity and storativity) using standard transient well analysis techniques;
- A best estimate of the hydraulic conductivity (K) equal to 4.0E-07 m/s was determined using a steady-state analytical solution. The estimate for K is believed to be too low given that drawdown was observed in a monitoring well located 26 feet from the pumping well, yet the linear velocity determined using the estimated K and site hydraulic gradient would not have predicted such a quick water level response. This implies that the entire thickness of the water-bearing zone may not be contributing substantially to the total flow to the well. Rather, preferential flow paths through thin water-bearing layers having much greater hydraulic conductivity is more plausible;
- Wellhead parameters including dissolved oxygen, temperature, specific conductance and pH stabilized early during the pumping well test suggesting samples collected and analyzed were representative of *in situ* conditions;

- Dissolved oxygen levels measured in groundwater samples collected from the pumping well were at or below 1 mg/L signifying reducing conditions. Anaerobic bacteria that degrade VOCs by reductive dechlorination favor such conditions;
- Groundwater samples collected during the test and analyzed for VOCs contained PCE and TCE that have been shown in the literature to be degraded by reductive dechlorination;

Analyses and observations resulting from the pumping well test indicated that injection of HRC[®] into the artificial fill was feasible. The quick hydraulic response observed during the test provided confidence that HRC[®] would disperse in the groundwater and travel within a reasonable time period to a downgradient observation point where the effects of biodegradation could be detected, if it occurred. Therefore, it was proposed to inject HRC[®] within 6-feet and upgradient from MW71b-98-13 where it would dissolve and travel with the groundwater degrading contaminants along the way. Samples collected from monitoring well MW71b-98-13 would be used to establish a baseline against which future analyses could be compared and the effectiveness of the bioremediation technology could be assessed.

SECTION 3

BASELINE SAMPLING

Groundwater samples were collected from monitoring well MW71b-98-13 prior to injection on Sept. 24, 2003 and Sept. 26, 2003. A peristaltic pump was used to extract groundwater from the monitoring well and to pump it through a flow-through cell where wellhead parameters were measured. A portable meter was used to measure the wellhead parameters including dissolved oxygen, temperature, pH and specific conductance. Groundwater samples were taken after the wellhead parameters stabilized and after a minimum of three-casing volumes were purged from the well. Samples were also collected and analyzed on site for dissolved carbon dioxide, ferrous iron, and total sulfide using field test kits.

The samples were analyzed for the chemical constituents summarized in Table 1 and the volatile organic compounds (VOCs) listed for EPA method 8260 summarized in Table 2. (Note that Table 2 only summarizes the EPA method 8260 constituents detected in the samples). Wellhead parameters are reported in Table 3. The following laboratories or field test kits were used to analyze the samples:

- Microseeps, Inc. (Pittsburgh, PA) analyzed the samples for total organic carbon (TOC), total inorganic carbon (TIC), volatile fatty acids (VFA), hydrogen, and light hydrocarbon gases (LHG including ethene, ethane, and methane).
- Berkeley Lab analyzed the samples for EPA method 8260 parameters.
- BC Laboratories, Inc. (Bakersfield, CA) analyzed the samples for the remaining constituents excluding carbon dioxide and ferrous iron.
- Dissolved carbon dioxide, ferrous iron, and sulfide were measured by field personnel on site during sampling using a Chemetrics K-1910 test kit and Hach test kits 26672-00 and 223801, respectively.

Unfortunately, the baseline VFA sample sent to Microseeps broke during shipping and the nitrate/nitrite sample sent to BC Laboratories exceeded their hold time. Therefore, a

second round of samples were collected from monitoring well SB71b-99-1 on Sept. 30, 2003 after HRC[®] was injected. The sample was collected from SB71b-99-1 because it was believed that HRC[®] had already reached well MW71b-98-13 and would, therefore, influence the baseline results had MW71b-98-13 been sample instead. Well SB71b-99-1 is located immediately upgradient from the HRC[®] injection location (Figure 2) and is connected hydraulically to MW71b-98-1 based on the pumping test results.

SECTION 4

HYDROGEN RELEASE COMPOUND (HRC®) INJECTION

Injection of HRC® into the formation was accomplished using a Geoprobe® rig, push rods, water bath, and a high-pressure pump. Vironex, Inc., located in San Leandro, California, was the contractor who provided the equipment and operators needed to perform the work. On-site LBNL personnel supervised the contractor. The injection process took about 5 hours and was started and completed on September 26, 2003.

Site preparation consisted of locating underground utilities including construction of a shallow 1.7 ft. wide, 8.4 ft. long, 4 ft. deep trench used to visually locate a 6-inch water main (Figure 3). Two nearby stormwater drains were covered with plastic and sand bags to prevent accidental spills of HRC® or drilling fluids from entering the drain. No spills occurred. Approximately 330 pounds of HRC® was purchased for the pilot study from Regenesys, Inc. headquartered in San Clemente, California and delivered to LBNL.

A Geoprobe® rig was used to advance push rods to a total depth of 25 to 30 ft. below ground level where the injection process began. Thirty pound plastic buckets containing HRC® were pre-heated in a water bath to about 130°F prior to injection to lower the viscosity of the honey-like material, allowing it to be easily pumped down hole through the hollow push rods. A high-pressure pump was used to inject the HRC® and was calibrated prior to use by counting the number of strokes required to pump about 4 pounds of HRC® into an empty bucket. Warm HRC® was then injected under pressure (240-300 pounds per square inch) through the push rods, out the open jets of the injection tool located at the bottom of the push rods and into the formation. The injection rods were pulled back one foot at a time and approximately 4 pounds of HRC® was injected per linear foot of boring. Injection was intentionally halted once the injection interval reached the top of the water table.

Table 4 summarizes the total amount of HRC[®] injected into each of the four Geoprobe[®] borings shown on Figure 3. The borings were installed in the order that they are numbered. As noted in the comment column of Table 4, a small quantity of HRC[®] was observed flowing out of the top of boring #2 into the trench when injecting into borings #3 and #4. This implies that a preferential flow path or short circuit through the formation developed between boring #2 and #3 and between #2 and #4. The short circuit between borings was observed when the injection interval reached a depth of 20 ft. in boring #3 and 15 ft. in boring #4. Broad vertical coverage of HRC[®] is still believed to have taken place given the relatively constant pressure observed during injection into individual one foot intervals.

SECTION 5

POST-INJECTION GROUNDWATER SAMPLING

Post-injection groundwater sampling was initiated after HRC[®] injection to determine the effectiveness of the treatment technology. Groundwater samples were collected from monitoring well MW71b-98-13 on a weekly to biweekly schedule for a period of 3 months, and monthly thereafter. Wellhead parameters including dissolved oxygen, temperature, and specific conductance were collected using a portable meter when the well was purged to ensure that representative groundwater samples were obtained. Weekly and biweekly samples were collected using a peristaltic pump and analyzed for VOCs (i.e., EPA method 8260 parameters). Summaries of the VOC analyses and wellhead parameters are summarized in Table 2 and 3, respectively. (Note that Table 2 summarizes only the VOCs detected in the samples).

An expanded list of analyses was performed on groundwater samples taken from MW71b-98-13 on a bimonthly schedule (Table 1). These analyses were conducted to supplement and enhance the information provided by the VOC analyses. The VOC analyses provide an overall view of the effectiveness of the treatment technology, while the expanded list provides a more detailed look at the hydrochemical conditions controlling the bioremediation process.

The following laboratories and field test kits were used to analyze the samples:

- a. Microseeps, Inc. (Pittsburgh, PA) analyzed the samples for total organic carbon (TOC), total inorganic carbon (TIC), volatile fatty acids (VFA), hydrogen, and light hydrocarbon gases (LHG including ethene, ethane, and methane).
- b. Berkeley Lab analyzed the samples for EPA method 8260 parameters; and
- c. BC Laboratories, Inc. (Bakersfield, CA) analyzed the samples for the remaining constituents (excluding carbon dioxide and ferrous iron) and some of the EPA method 8260 samples.

- d. Dissolved carbon dioxide, ferrous iron, and sulfide were measured by field personnel on site during sampling using a Chemetrics K-1910 test kit and Hach test kits 26672-00 and 223801, respectively.

SECTION 6

INTERPRETATION OF PILOT TEST RESULTS

The baseline samples, historical results from previous quarterly and annual sampling events, and post-injection analytical results (Tables 1 through 3) provide the basis for evaluating the pilot test results and assessing the continued effectiveness of enhanced bioremediation. The criteria given in Table 5 provide the basis for interpreting the analytical results.

The electron acceptors and geochemical parameters listed in Table 1 provide the basis for measuring the potential success of CAHs degradation by reductive dechlorination and monitoring of site-specific conditions that can lead to its arrest. Based on thermodynamic theory, reductive dechlorination of PCE, TCE and cis-1,2-DCE will not proceed until electron acceptors including oxygen, nitrate, manganese (IV), and ferric iron have been reduced (or are not present). Microorganisms who utilize these electron acceptors during respiration will flourish and dominate over species that would otherwise utilize the CAHs as their primary electron acceptors. Oxygen, nitrate, and dissolved manganese concentration levels are nondetect or very low (Table 1) implying these electron acceptors are not available to compete with the CAHs. Ferric iron (Fe^{3+}) is perhaps the most important of the natural electron acceptors to be considered when evaluating the redox potential and microbial respiration processes that can lead to reductive dechlorination of CAHs. The detection of ferrous iron (Fe^{2+} , the reduced form of ferric iron, Fe^{3+}) reported in Table 1 is a very strong indicator that redox conditions, which promote reductive dechlorination of CAHs, exist in the core of the groundwater plume at Building 71b. Post-injection electron acceptor data (Table 1) and the interpretation of these data (based on the criteria in Table 5), indicates that redox conditions that support reductive dechlorination have not changed significantly throughout the 6-month pilot test following HRC[®] injection.

Even though redox conditions that favor CAH degradation may be present, hydrogen ions must also be readily available as a substitute for chlorine in the CAH structure. In addition, a carbon source must be available to act as the electron donor (i.e., food). HRC[®] provides both of these components. Groundwater pH (Table 3) dropped dramatically within a few days of injecting HRC[®] signifying that the HRC[®] had begun to hydrate and release volatile fatty acids in the groundwater, as expected. This was confirmed later on December 4, 2003 when volatile fatty acids (lactic, pyruvic, acetic acids, etc.) were detected in the first scheduled round of groundwater samples collected for this purpose (Table 1). Analytical data from this same round of samples also provide ample evidence that dissolved hydrogen and total organic carbon levels rose dramatically above background levels (Table 1) within 2 months of injecting HRC[®] into the water-bearing zone. The latest round of analyses from the February 19, 2004 sampling indicates that dissolved hydrogen and total organic carbon levels have since decreased and the relative abundance of volatile fatty acids has shifted from lactic acid to acetic acid as hydrogen is released. This may imply that the HRC[®] is “aging” and will become less effective over time as volatile fatty acids decrease (releasing smaller amounts of hydrogen) and as carbon is consumed.

The data described above demonstrates that redox conditions are favorable, and that sufficient electron donors (in the form of total organic carbon and volatile fatty acids) are present, to support microbial respiration. In addition, indicator parameters including pH, volatile fatty acids, and dissolved hydrogen, show that hydrogen ions are present in the groundwater and potentially available to support substitution for chlorine in the CAH structure. The question that remains is whether microorganisms that degrade the CAHs are present, or whether they are competing with other organisms that utilize more readily available electron acceptors in the respiration process? Indirect evidence that these bacteria are present can be concluded from the observed decrease in contaminant concentration data reported in Table 2 and large increase in metabolic byproducts of microbial respiration (i.e., ethene, ethane, and methane) reported in Table 1. The PCE and TCE concentrations detected in samples from monitoring well MW71b-98-13 decreased immediately following HRC[®] injection on September 26, 2003 as shown on

Figure 4 to levels approaching the cleanup standard for drinking water. In contrast, cis-DCE, and to a lesser extent, VC concentrations have increased. Cis-DCE and VC are known byproducts of the reductive dechlorination process and may increase as PCE and TCE are biodegraded to cis-DCE, VC and eventually ethene, ethane, methane, carbon dioxide and water.

Approximately 3 to 5 weeks after HRC[®] injection, both the cis-DCE and VC concentrations began to drop and were at or below historical concentrations by mid December 2003 (Table 2). Starting in mid January 2004, cis-DCE concentrations began to climb. This is attributed to seasonal rainfall and subsequent recharge that has flushed PCE and TCE out of soils causing contaminant levels to rise in groundwater samples collected from the upgradient source area (near well MW71B-99-3R, Figure 2). This leads us to believe that higher concentrations of PCE and TCE, flowing downgradient from the source with the groundwater, are being swept through the treatment area and degraded by the HRC[®] increasing the cis-DCE levels at MW71b-98-13. Meanwhile, PCE and TCE concentrations have remained relatively constant at MW71b-98-13 implying that enhanced bioremediation of these compounds is quite effective. Reductive dechlorination of cis-DCE and VC appears to be less effective in the short-term, but given time they also appear to degrade.

SECTION 7

CONCLUSIONS

The degradation of PCE and TCE, and to a lesser extent cis-DCE and VC, using HRC[®]-enhanced natural bioremediation is a feasible remedial technology for the Building 71b core groundwater contaminant plume. Natural conditions appear to favor the use of this technology, producing a 10-fold reduction in PCE and TCE to levels meeting regulatory standards (Table 3, maximum contaminant level [MCL]) and 3-fold decrease in cis-DCE contaminant concentrations within 6 months. Vinyl chloride, which is known to be more recalcitrant to biodegradation under anaerobic conditions, has remained relatively stable within its historical range of concentration values.

The introduction of HRC[®] into the water-bearing zone at Building 71b has not been without negative consequences. The groundwater has developed a strong septic-like odor that is likely caused by the decaying sugar and organic acids found in the HRC[®]. Introduction of large amounts of organic material has created conditions similar to a shallow house-hold septic field, where biological degradation of the organic waste can impart an odor and impact the taste of shallow groundwater. Unlike a septic system, however, HRC[®] does not introduce pathogens found in human or animal wastes, but simply promotes the growth of indigenous microorganisms that can degrade the contaminants. Given the likelihood that the shallow water-bearing zone beneath Building 71b will not be used as a potable water supply, quick and effective mitigation of site contaminants using enhanced bioremediation is clearly protective of public health and safety; whereas, the impact on taste and odor should be viewed as a tradeoff, or cost, of obtaining this benefit, should this technology be used as the final remedy.

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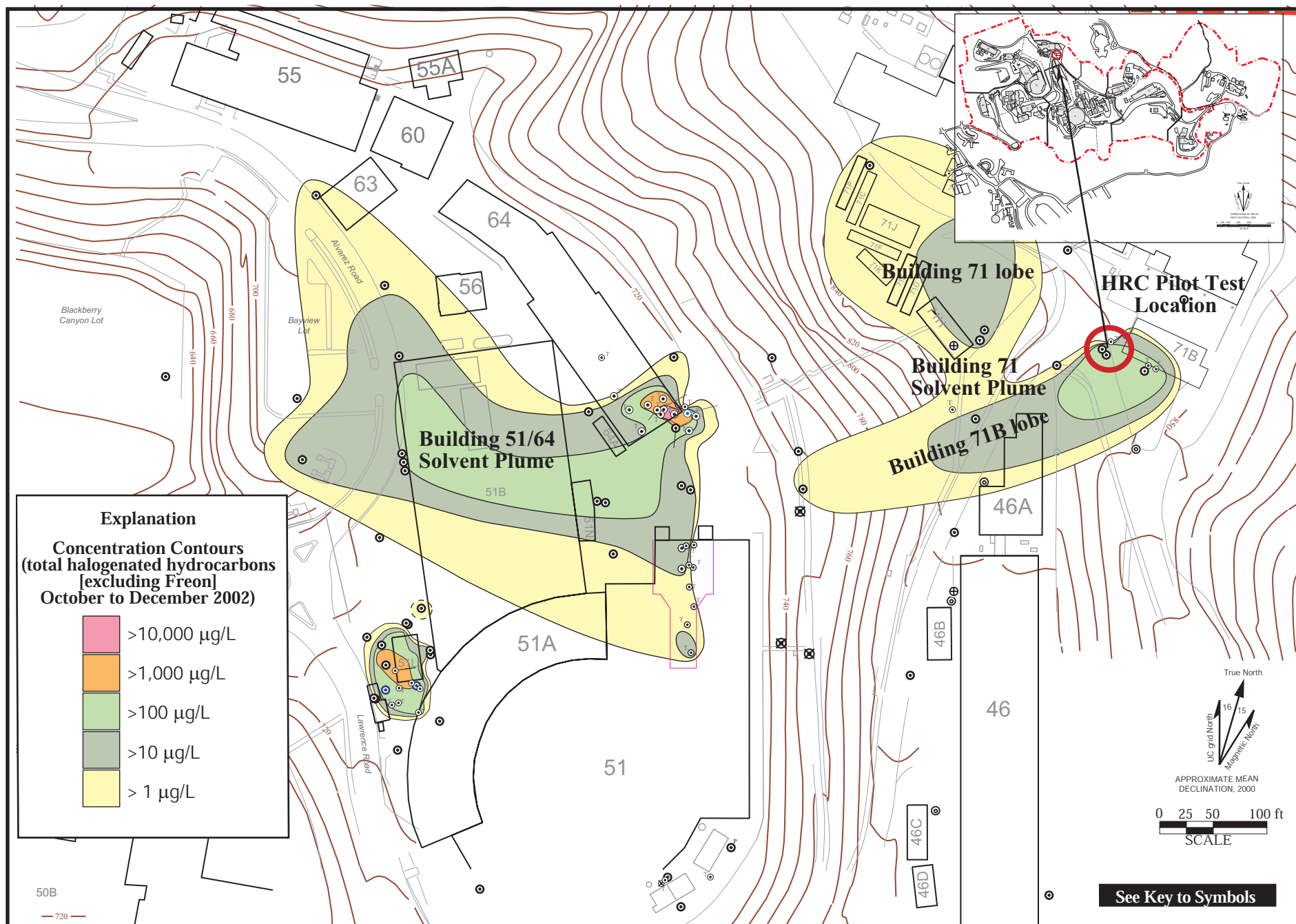


Figure 1. Location of Building 71b HRC Pilot Test.

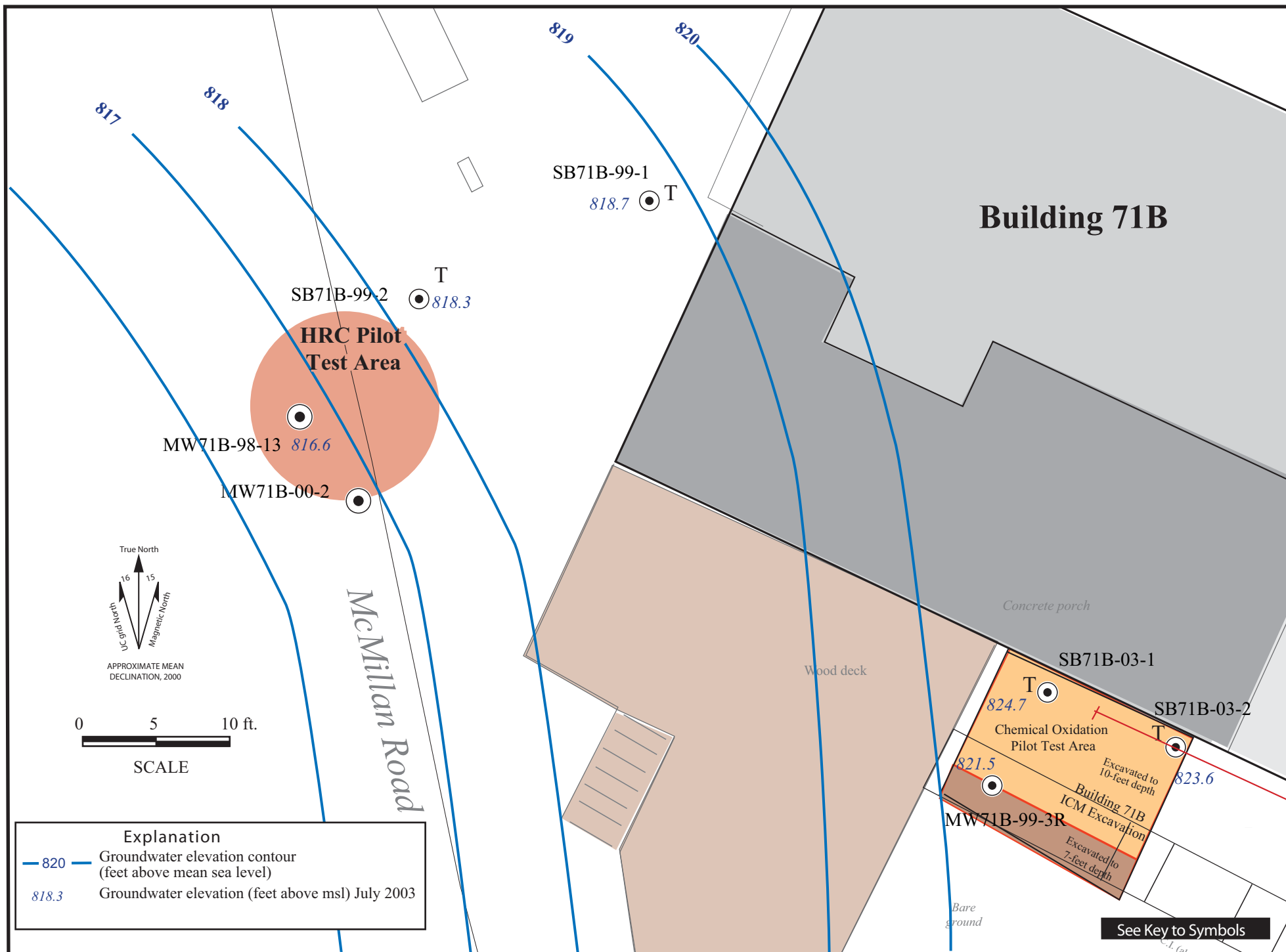


Figure 2. Site Map Building 71b HRC Pilot Test.

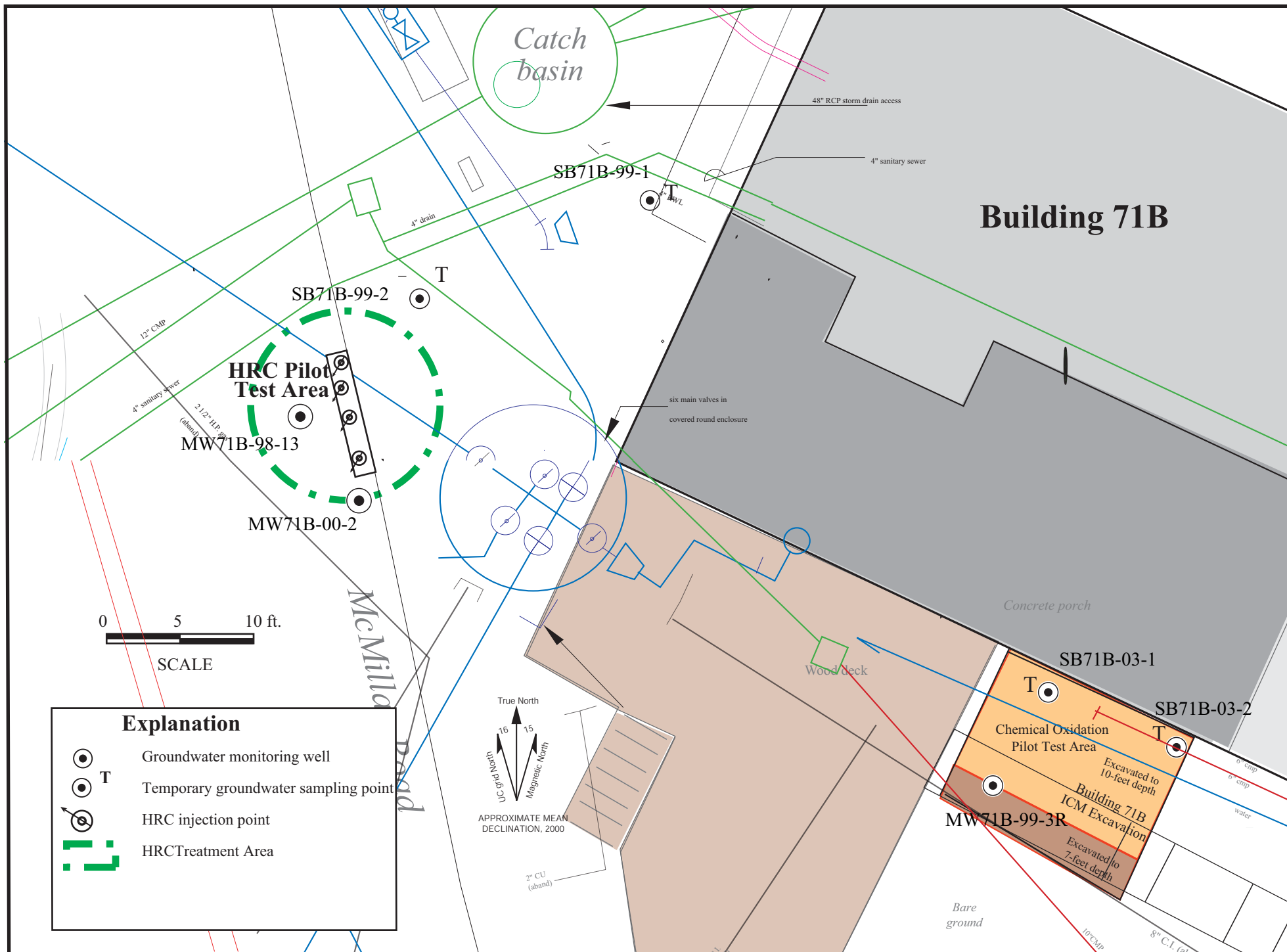


Figure 3. HRC Injection Points.

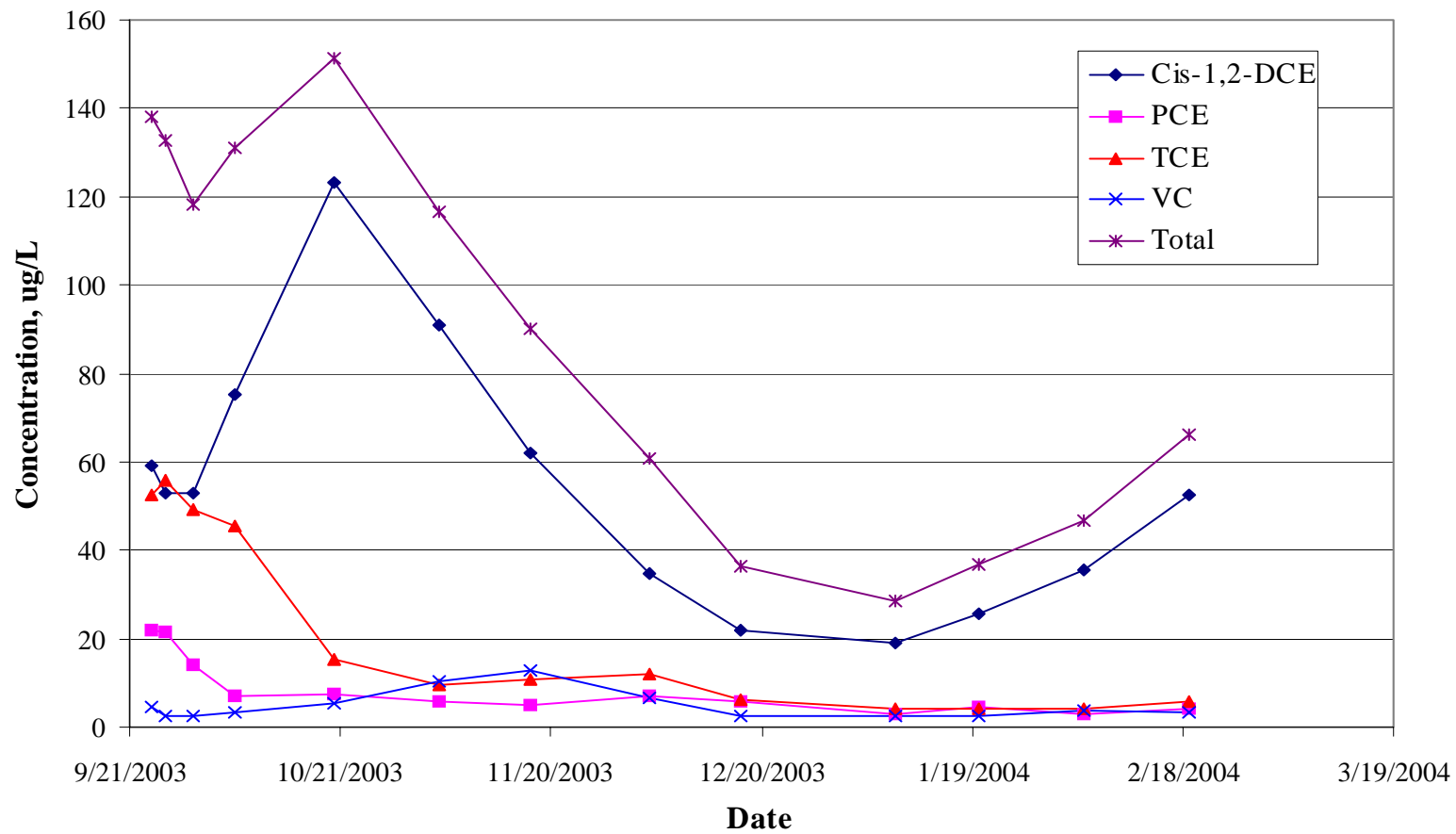


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Table 1. Analytical Results for Electron Acceptors, Metabolic Byproducts, and Other Indicator Parameters

| Analyte | Baseline | | 4-Dec-03 | 19-Feb-04 | Unit |
|--|--------------------------|-------------------------|-------------|-------------|------|
| | MW71b-98-13 ^a | SB71b-99-1 ^b | MW71b-98-13 | MW71b-98-13 | |
| DO | 0.3 | 0.97 | 0.50 | 0.50 | mg/L |
| Nitrate as NO ₃ ⁻ | – | <1.0 | <0.88 | <0.88 | mg/L |
| Nitrite as NO ₂ | – | <1.0 | 0.11 | <0.065 | mg/L |
| Total Manganese | – | – | 25 | 44 | mg/L |
| Dissolved Mn | 2.4 | 2.5 | – | – | mg/L |
| Ferrous Iron (Fe ²⁺) | 4.0 ^c | – | 5.2 | 4.8 | mg/L |
| Sulfate as SO ₄ ²⁻ | 17 | 18 | 34 | 5.0 | mg/L |
| Total Sulfide | <0.1 | – | <0.1 | <0.1 | mg/L |
| Ethene | 89 | – | 3600 | 2000 | ng/L |
| Ethane | 72 | – | 220 | 44 | ng/L |
| Methane (CH ₄) | 53 | – | 1100 | 3600 | μg/L |
| Carbon Dioxide (CO ₂) | <10 | – | <10 | <10 | mg/L |
| Alkalinity as CaCO ₃ | 240 | 260 | 1400 | 2000 | mg/L |
| TIC (as CaCO ₃) | 790 | – | 3400 | 2700 | mg/L |
| TOC | <5.0 | – | 7200 | 2100 | mg/L |
| Hydrogen | 0.79 | – | 20000 | 36 | nM |
| Acetic Acid | – | <0.070 | 187 | 415 | mg/L |
| Butyric Acid | – | <0.070 | < 70 | 5300 | |
| Lactic Acid and HIBA | – | <0.070 | 1930 | 860 | mg/L |
| Pentanoic Acid | – | <1.0 | 109 | 216 | mg/L |
| Propionic Acid | – | <0.070 | 870 | 1370 | mg/L |
| Pyruvic Acid | – | <0.070 | 3100 | < 70 | mg/L |
| Chloride (Cl) | 12 | 11 | 74 | 38 | mg/L |
| Total Iron | – | – | 25 | 100 | mg/L |
| Dissolved Iron | 0.74 | – | – | – | mg/L |

^a Sample was collected on 9/24/03 prior to HRC[®] injection.

^b Sample was collected on 9/30/03 from upgradient well SB71b-99-1 after HRC[®] injection because original baseline sample was broken during transport or exceeded hold time.

^c Sampled and analyzed for Fe²⁺ on 10/6/03.

“–” Indicates sample was not analyzed for this constituent.

Table 2. Summary of Analytical Results for Volatile Organic Compounds (concentrations in µg/L).

| Constituent | MCL | 6/25/99 | (D) | 9/15/99 | 11/16/99 | 3/7/00 | 5/18/00 | 6/20/00 | 9/11/00 |
|--------------------------------|------------|-----------------|----------------|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|--------------------------------------|
| 1,1 Dichloroethene | 6 | <1 | 1.1 | <1 | <1 | <1 | <1 | 1.0 | |
| Cis-1,2-Dichloroethene | 6 | 58.0 | 45.0 | 47.5 | 61.1 | 51.6 | 65.8 | 43.0 | 53.4 |
| Trans-1,2-Dichloroethene | 10 | <1 | 0.57 | <1 | <1 | <1 | <1 | 1.5 | |
| Tetrachloroethene | 5 | 146.0 | 180.0 | 90.1 | 112.0 | 36.1 | 104.0 | 210.0 | 171.9 |
| Trichloroethene | 5 | 86.4 | 91.0 | 81.4 | 79.6 | 50.4 | 120.0 | 100.0 | 119.3 |
| Vinyl Chloride | 0.5 | 2.6 | 4.0 | 3.9 | 3.7 | 7.7 | 3.8 | 2.9 | 3.0 |
| Total Halogenated Hydrocarbons | | 293 | 322 | 223 | 256 | 146 | 294 | 358 | 347.6 |
| Constituent | MCL | 11/29/00 | 3/19/01 | (D) | 5/22/01 | 9/6/01 | 11/20/01 | 2/28/02 | 5/16/02 |
| 1,1 Dichloroethene | 6 | <1 | 1.1 | 0.98 | <1 | <1 | 1.2 | <1 | <1 |
| Cis-1,2-Dichloroethene | 6 | 89.2 | 91.1 | 71.0 | 68.0 | 65.5 | 46.9 | 58.7 | 81.0 |
| Trans-1,2-Dichloroethene | 10 | <1 | <1 | 1.8 | <1 | <1 | <1 | <1 | <1 |
| Tetrachloroethene | 5 | 68.8 | 131.8 | 120.0 | 149.7 | 106.8 | 102.8 | 121.9 | 130.1 |
| Trichloroethene | 5 | 128.4 | 85.1 | 77.0 | 71.8 | 88.0 | 68.0 | 90.5 | 88.7 |
| Vinyl Chloride | 0.5 | 6.5 | 5.8 | 3.4 | 16.0 | 5.3 | 2.8 | 5.9 | 5.3 |
| Total Halogenated Hydrocarbons | | 293 | 315 | 274 | 306 | 266 | 222 | 277 | 305 |
| Constituent | MCL | 9/3/02 | 2/12/03 | 8/5/03^b 11:07 | 8/7/03^b 11:07 | 8/7/03^b 12:07 | 8/7/03^b 15:00 | 8/8/03^b 11:00 | 8/11/03^b 11:00 |
| 1,1 Dichloroethene | 6 | <1 | <1 | <1 | <1 | <1 | <1 | <1 | <1 |
| Cis-1,2-Dichloroethene | 6 | 74.9 | 63.8 | 57.3 | 50.5 | 55.9 | 76.7 | 80.1 | 84.1 |
| Trans-1,2-Dichloroethene | 10 | <1 | <1 | <1 | <1 | <1 | <1 | <1 | <1 |
| Tetrachloroethene | 5 | 75.3 | 115.0 | 143.0 | 49.1 | 42.1 | 39.1 | 72.8 | 50.0 |
| Trichloroethene | 5 | 71.0 | 89.8 | 78.1 | 54.7 | 70.2 | 79.1 | 89.0 | 79.8 |
| Vinyl Chloride | 0.5 | 4.1 | 5.2 | 2.9 | 2.2 | 2.0 | 3.6 | 3.6 | 4.3 |
| Total Halogenated Hydrocarbons | | 225.3 | 274 | 281.3 | 156.5 | 170.2 | 198.5 | 245.5 | 218.2 |

Table 2. Summary of Analytical Results for Volatile Organic Compounds (concentrations in µg/L) (cont'd.)

| Constituent | MCL | 9/24/03^a | 9/26/03^a | 9/30/03 | 10/6/03 | 10/20/03 | 11/4/03 | 11/17/03 | 12/4/03 |
|--------------------------------|------------|----------------------------|----------------------------|----------------|----------------|-----------------|----------------|-----------------|----------------|
| 1,1 Dichloroethene | 6 | <1 | <1 | <1 | <1 | <1 | <1 | <1 | <1 |
| Cis-1,2-Dichloroethene | 6 | 59 | 53.1 | 53 | 75.2 | 123 | 91 | 61.9 | 34.6 |
| Trans-1,2-Dichloroethene | 10 | <1 | <1 | <1 | <1 | <1 | <1 | <1 | <1 |
| Tetrachloroethene | 5 | 21.8 | 21.4 | 14 | 7.2 | 7.6 | 5.6 | 4.8 | 7.2 |
| Trichloroethene | 5 | 52.7 | 55.7 | 49 | 45.4 | 15.4 | 9.6 | 10.6 | 12 |
| Vinyl Chloride | 0.5 | 4.4 | 2.5 | 2.4 | 3.3 | 5.3 | 10.3 | 12.9 | 6.8 |
| Total Halogenated Hydrocarbons | | 137.9 | 132.7 | 118.4 | 131.1 | 151.3 | 116.5 | 90.2 | 60.6 |
| Constituent | MCL | 12/17/03 | 1/8/04 | 1/20/04 | 2/4/04 | 2/19/04 | | | |
| 1,1 Dichloroethene | 6 | <1 | <1 | <1 | <1 | <1 | | | |
| Cis-1,2-Dichloroethene | 6 | 22 | 19.2 | 25.8 | 35.6 | 52.7 | | | |
| Trans-1,2-Dichloroethene | 10 | <1 | <1 | <1 | <1 | <1 | | | |
| Tetrachloroethene | 5 | 5.8 | 2.8 | 4.6 | 2.8 | 4.1 | | | |
| Trichloroethene | 5 | 6.4 | 4.1 | 4.2 | 4.3 | 5.9 | | | |
| Vinyl Chloride | 0.5 | 2.3 | 2.5 | 2.3 | 3.9 | 3.4 | | | |
| Total Halogenated Hydrocarbons | | 36.5 | 28.6 | 36.9 | 46.6 | 66.1 | | | |

^a Baseline Samples – treatment with HRC[®] began on 9/26/03 after the 9/26/03 sample was collected.

^b 8/7 – 8/11/03 samples were collected during pumping well test.

(D) = duplicate sample.

Table 3. Summary for Wellhead Parameters.

| Wellhead Parameter | Baseline 9/24/03 | 9/29/03 | 9/30/03 | 10/1/03 | 10/2/03 | 10/4/03 | 10/6/03 | 10/14/03 | 10/20/03 |
|------------------------------|-----------------------------|-----------------|-----------------|----------------|-----------------|-----------------|----------------|-----------------|-----------------|
| | | | | | | | | | |
| DO (mg/L) | 0.3 | 0.37 | 0.22 | 0.48 | 0.22 | 0.23 | 0.26 | 0.31 | 0.71 |
| Temperature (°C) | 17.4 | 19.1 | 16.3 | 17.9 | 16.8 | 15.9 | 17.8 | 17.2 | 17.0 |
| pH | 6.01 | 2.63 | 5.12 | 4.92 | 5.66 | 5.42 | 5.47 | 4.24 | 5.19 |
| Specific conductance (µmhos) | 698 | 1310 | 1100 | 1180 | 1180 | 2370 | 1446 | 2050 | 2330 |
| Wellhead Parameter | 10/27/03 | 11/12/03 | 11/17/03 | 12/4/03 | 12/11/03 | 12/17/03 | 1/8/04 | 1/20/04 | 2/4/04 |
| | | | | | | | | | |
| DO (mg/L) | 0.33 | 0.14 | 0.33 | 0.50 | 0.58 | 0.53 | 0.45 | 0.46 | 0.45 |
| Temperature (°C) | 22.7 | 19.5 | 17.4 | 14.5 | 14.1 | 14.7 | 16.8 | 13.9 | 18.4 |
| pH | 4.62 | 5.05 | 5.23 | 4.8 | 5.36 | 5.23 | 5.24 | 5.26 | 5.30 |
| Specific conductance (µmhos) | 2460 | 3010 | 2850 | 3280 | 2920 | 2630 | 2190 | 2680 | 3310 |
| Wellhead Parameter | 2/19/04 | | | | | | | | |
| DO (mg/L) | 0.50 | | | | | | | | |
| Temperature (°C) | 14.8 | | | | | | | | |
| pH | 5.34 | | | | | | | | |
| Specific conductance (µmhos) | 3110 | | | | | | | | |

Table 4. Summary of HRC[®] Injection

| Boring No. | Injection Depth Below Ground Level (ft) | Total HRC[®] Injected (lbs.) | Comment |
|-----------------------------------|--|--|---|
| 1 | 12-30 | 80 | HRC [®] returned to surface |
| 2 | 12-30 | 110 | No return |
| 3 | 12-30 | 80 | Injection into #3 forced HRC [®] out of boring #2 |
| 4 | 15-25 | 30 | Injection into #4 forced HRC [®] out of boring #2 |
| Total HRC Injected (lbs.): | | 300 | |

Table 5. Analytical Parameters and Trends in Concentrations During Natural Biodegradation.

| Analysis | Terminal Electron Accepting Process | Trend in Analyte Concentration During Natural Biodegradation | Possible Concentration in Most Contaminated Zone | Significance |
|---|---|--|--|--|
| Electron Acceptors and Metabolic Byproducts | | | | |
| Dissolved Oxygen (DO) | Aerobic Respiration | Decreases | > 0.5 mg/L | Aerobic conditions; suppresses the reductive dechlorination pathway at higher concentration. |
| | | | > 1.0 mg/L | Vinyl Chloride may be oxidized aerobically |
| Nitrate (NO ₃ ⁻) | Denitrification | Decreases | < 1.0 mg/L | At higher concentrations, may compete with reductive dechlorination pathways. |
| Nitrite (NO ₂ ⁻) | Denitrification | Increases | | Produced as an intermediate byproduct during denitrification. |
| Manganese (Mn ²⁺) | Manganese IV Reduction | Increases | > 1.0 mg/L | Reductive dechlorination pathway possible |
| Ferrous (II) Iron (Fe ²⁺) | Ferric (III) Iron (Fe ³⁺) Reduction | Increases | > 1.0 mg/L | Reductive dechlorination pathway possible |
| Sulfate (SO ₄ ²⁻) | Sulfate Reduction | Decreases | < 20 mg/L | At higher concentrations, may compete with reductive dechlorination pathways. |
| Sulfide (H ₂ S/HS ⁻) | Sulfate Reduction | Increases | > 1 mg/L | Reductive dechlorination pathway possible |
| Methane (CH ₄) | Methanogenesis | Increases | > 0.5 mg/L | Ultimate reductive daughter produce; vinyl chloride may accumulate. |
| | | | < 0.5 mg/L | Vinyl choride may be oxidized aerobically |
| Other Indicator Parameters | | | | |
| Alkalinity | All processes listed above | Increases | > 2 times background | Results from interaction of carbon dioxide with aquifer materials. |
| Carbon Dioxide (CO ₂) | All processes listed above | Increases | > 2 times background | Ultimate oxidative daughter product |
| Chloride (Cl) | Reductive dechlorination or direct oxidation of chlorinated compounds | Increases | > 2 times background | Daughter product of reductive dechlorination pathways. |

Table 5. Analytical Parameters and Trends in Concentrations During Natural Biodegradation (cont'd.)

| Analysis | Terminal Electron Accepting Process | Trend in Analyte Concentration During Natural Biodegradation | Possible Concentration in Most Contaminated Zone | Significance |
|---|---|--|--|---|
| <i>Other Indicator Parameters (cont'd.)</i> | | | | |
| Hydrogen | Denitrification, ferric iron reduction, sulfate reduction | Increases | > 1 nM/L | Reductive pathway possible: vinyl chloride may accumulate. |
| | | | < 1 nM/L | Vinyl chloride oxidized. |
| Oxidation-Reduction Potential (ORP) | All processes listed above | Decreases | < 50 mV | Reductive dechlorination pathway or anaerobic biodegradation possible. |
| | | | < -100 mV | Reductive dechlorination pathway likely |
| pH | — | | 5 < pH < 9 | Optimal range for reductive pathway. |
| | | | < 5 or > 9 | Outside optimal range for reductive pathway. |
| Temperature (°C) | — | | > 20°C | Biochemical process is accelerated. |
| Total Dissolved Inorganic Carbon (DIC) | — | — | — | Primary end product of aerobic microbial activity – measure of total microbial activity. |
| Total Organic Carbon (TOC) | — | | > 20 mg/L | Carbon and energy source; drives dechlorination; can be natural or anthropogenic. |
| Volatile Fatty Acids | Source of H used in reductive dechlorination | Increases | > 0.1 mg/L | Intermediate products resulting from biodegradation of aromatic compounds; serve as a carbon and energy source. |

Appendix A

MW71b98-13 Pumping Well Test

Building 71B Pumping Well Test

A short duration pumping well test was performed at Building 71B to characterize the water-bearing zone penetrated by monitoring well MW71b-98-13. This short report summarizes and interprets the results of hydraulic data, wellhead measurements, and water quality results obtained during the test.

Hydraulic Response

Groundwater was pumped from 98-13, a 2-inch diameter monitoring well that is completed in artificial fill with an open sand-pack interval lying 15 to 30 feet below ground level (bgl). Initial static water levels in 98-13 prior to pumping were 14.14 ft below top of casing (TOC). In comparison, the pre-pumping static water level in adjacent monitoring well MW71B-00-2, completed in the underlying Orinda formation, was 46.05 ft below TOC, which is below the fill. The sand pack for well 00-2 is from 45 to 60 ft. bgl.

A peristaltic pump was used to extract groundwater from monitoring 98-13 starting on 8/7/03 at 11:02 A.M. and ending 4 days later on 8/11/03 at 11:02 A.M. The cumulative volume of water produced from the well was periodically recorded during the test and was used to calculate the pumping rate. The pumping rate did not stabilize during the test (Figure 1), which is typical of low yield formations.

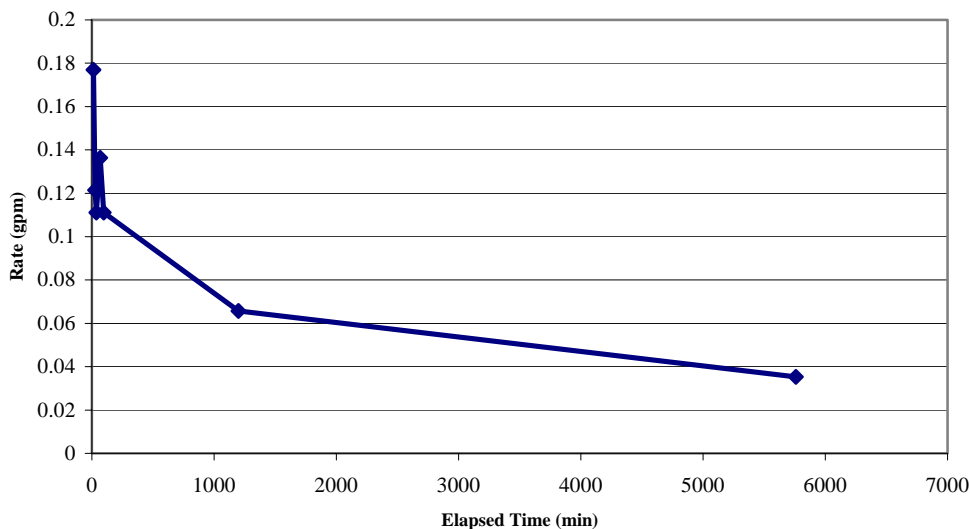


Figure 1. Extraction rate from pumped well MW71b-98-13

Water levels were measured in the pumping well and nearby observation wells 00-2, SB71b-99-1 and SB71b-99-2 using Omega MicroDAQ dataloggers and Druck pressure transducers. Depth to water level measurements were also periodically made by hand using an electric water level tape or probe. Temporary monitoring wells 99-1 and 99-2 are both completed in the fill with sand pack intervals ranging from 13-25 ft. and 8-20 ft. bgl, respectively.

Water level measurements in the pumping and observation wells were converted to hydraulic head elevations above mean sea level (Figure 2). The initial heads measured in monitoring wells 98-13, 99-1, and 99-2 were within 0.5 ft. of each other (818.19 to 818.69 ft.) prior to groundwater extraction. In comparison, the head measured in 00-2 was 32 feet, much lower than the other wells implying the two groups of wells (98-13, 99-1, and 99-2 versus 00-2) represent hydraulic responses from two different water-bearing zones – one zone being in the fill and the other in the Orinda formation. Further evidence leading to this conclusion is established by the fact that the water levels in monitoring wells 99-1 and 99-2 (Figure 3 and 4) decreased in response to pumping 98-13 (Figure 5) as expected, whereas water levels increased in 00-2 (Figure 6) during groundwater extraction. The very large difference in heads exhibited by the two water bearing-zones may also signify that the water in the upper fill layer may be perched.

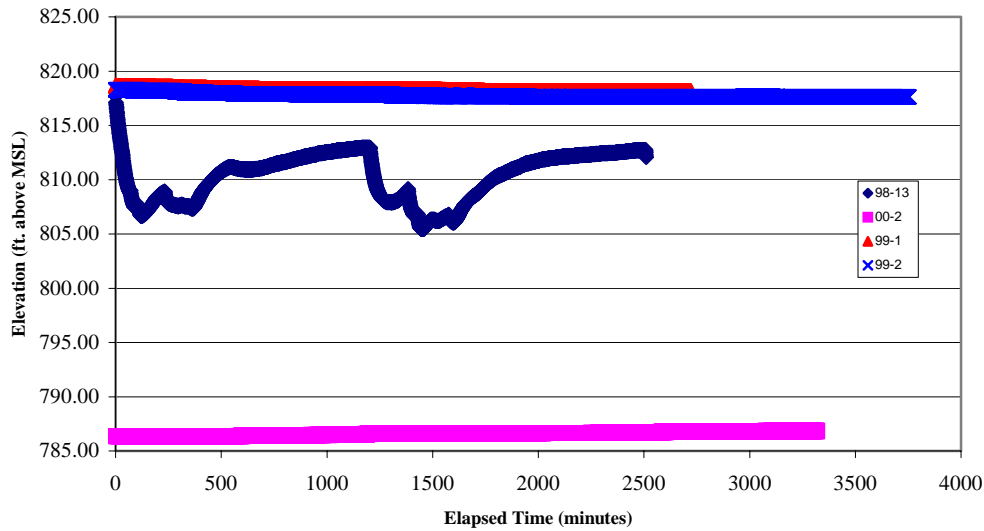


Figure 2. Water elevations in wells from start of pumping

Drawdown responses in the water table caused by pumping (Figure 3 through 5) were examined for obvious trends that would be amenable to evaluation using standard type-curve data matching techniques. However, given that the rates and heads were changing throughout the test (Figure 1 and 2), it was decided that the boundary conditions were not suitable for this type of analysis. In addition, sufficient data were not collected in order for the drawdown curves to developed a unique or distinct shape (i.e., signature typical of a Theis or delayed yield response) before the batteries powering the pressure transducers drained 2 to 3 days into the test. Instead, a simple first order estimate of the hydraulic conductivity (K) of the fill was calculated using the final pumping rate and heads measured in wells 99-1 and 99-2 and the steady-state solution for an confined aquifer given below:

$$K = \frac{Q}{2b\pi(h_2 - h_1)} \ln \frac{r_2}{r_1} \quad (1)$$

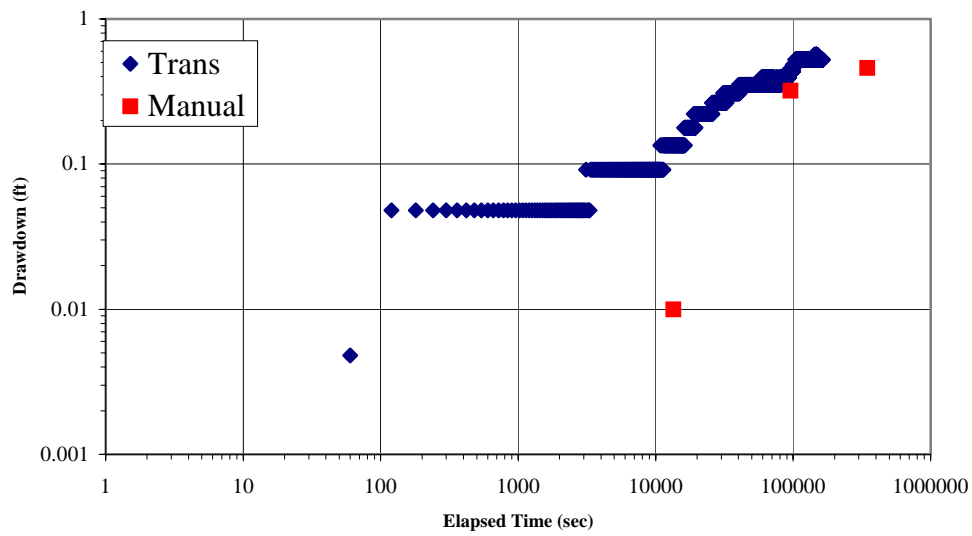


Figure 3. Drawdown response in monitoring well SB71b-99-1.

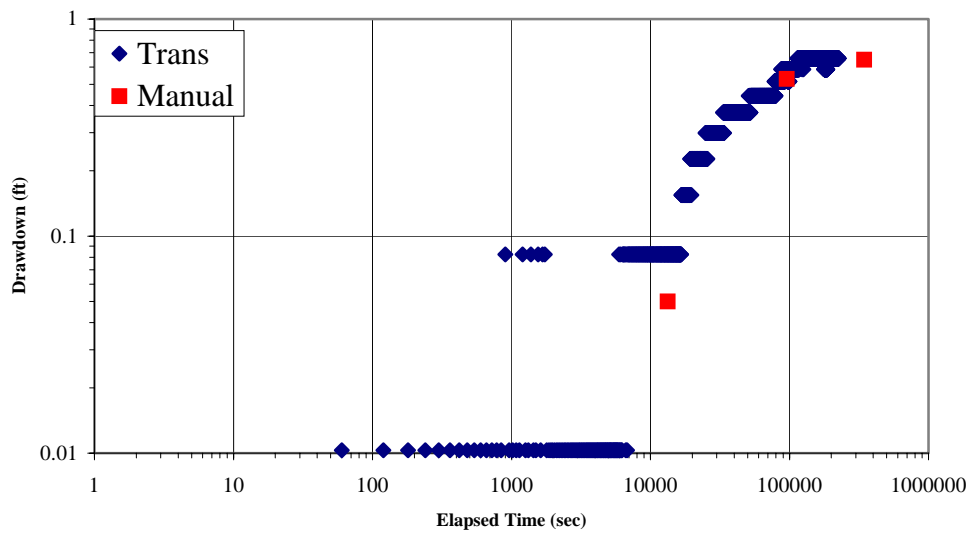


Figure 4. Drawdown response in monitoring well SB71b-99-2.

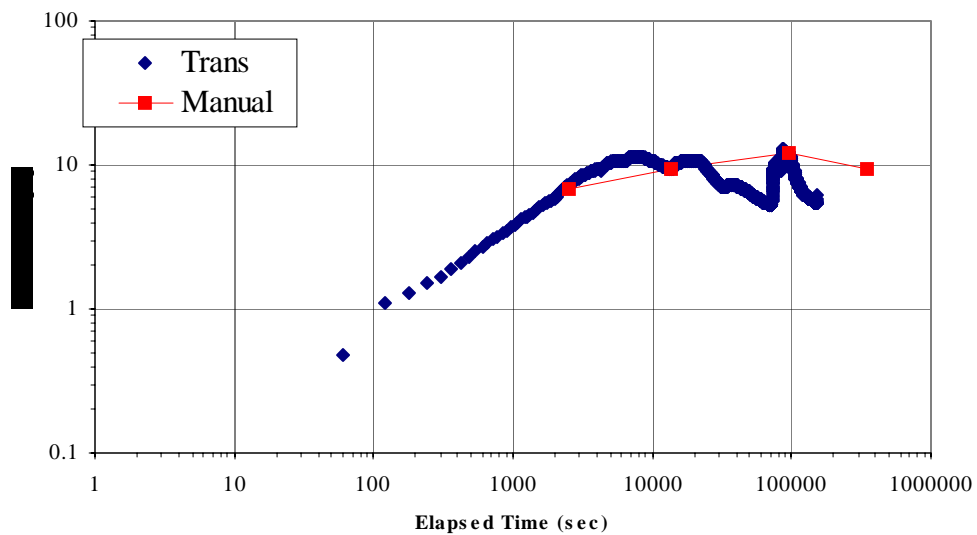


Figure 5. Drawdown response in pumping well MW71b-98-13.

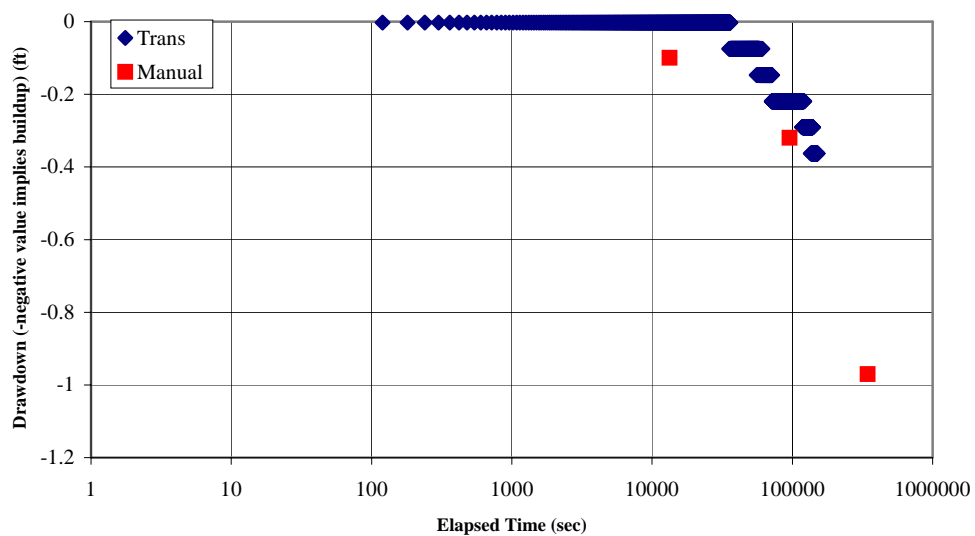


Figure 6. Buildup in well MW71b-00-2 during pumping.

where Q is the volumetric pumping rate (0.0353 gpm), b is the saturated thickness of the confined interval (assumed to be equal the sand-pack interval, i.e., ~12 ft.), r_1 and r_2 are the radial distances from the pumping well to observation well 99-2 (~12 ft.) and 99-1 (~26 ft.), and h_1 and h_2 are the head values at well 99-2 (817.62 ft.) and 99-1 (818.23 ft.), respectively. Using the values observed from the pumping test, K was estimated to be 0.85 gal/day/ft² (4.0E-07 m/s), which is within the hydraulic conductivity range for a silty sand (Freeze and Cherry, 1979, Table 2.3).

The derived K -value (i.e., 4.0E-07 m/s) is much smaller than would be expected to affect a drawdown response in the two nearby observation wells. Using an estimate of formation porosity of 20% and a steep hydraulic gradient of 0.1 m/m, the background linear groundwater velocity (without pumping) would only be on the order of 0.02 m (2 cm) per day. This estimate for velocity would only be marginally improved by increasing the gradient due to pumping to 1 m/m. Either the approach used to derive K underestimates its value by 1 to 2 orders of magnitude, implying that the entire sand pack thickness may not be contributing to flow to the well (i.e., preferential flow paths exist), or the porosity is much smaller than 20%. Given the extent and age of the contaminant plume, the contaminant velocity would suggest that the approach used to derive K is the source of error rather than the porosity.

Wellhead Parameters

Wellhead parameters including dissolved oxygen (DO), specific conductance, temperature and pH were measured in groundwater samples periodically collected during the test. A flow-through cell was placed in-line on the discharge line from the well and a calibrated YSI multi-purpose probe was used to measure the wellhead parameters in real time (Figure 7).

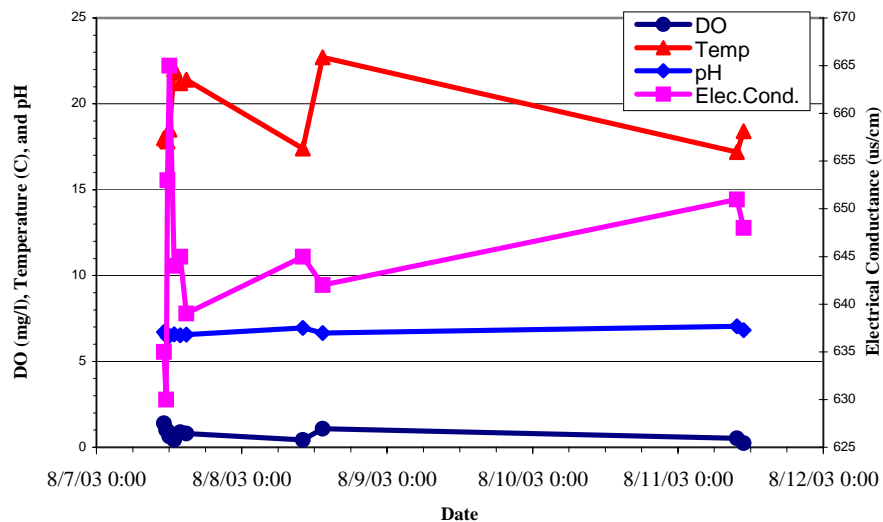


Figure 7. Wellhead parameters including DO, specific conductance, temperature and pH.

The wellhead parameters are normally collected during groundwater sampling events to determine how effective the well purging process is in obtaining groundwater samples from the formation that are representative of true aquifer conditions. The wellhead parameters typically stabilize after stagnant water in the well is purged and replaced by “fresh” groundwater flowing into the well from the formation. The DO and pH were found to be quite stable throughout the entire pumping test (Figure 7, Table 1). The specific conductance stabilized within an hour of the start of pumping and, in general, exhibited a small increase (10-12 $\mu\text{S}/\text{cm}$) thereafter through the end of the test. The groundwater temperature changed by 4 to 5°C, which is unusual and significant; however, the time of measurement appears to have influence the temperature readings more than any likely variation in temperature attributed to the formation. Note that the large groundwater temperatures (Figure 7, Table 1) occur in the afternoon when ambient air temperatures are also expected to be highest suggesting that the afternoon water temperatures were likely influenced by the heat and corresponding temperature of the test equipment (i.e., discharge line, flow through cell, and probe) used to make the measurement. Comparison of the temperature data collected only in the morning (Table 1) suggests that the temperatures were very stable throughout the pumping test (17.2 to 18.4°C). Note that the electrical conductance also exhibits an inverse correlation with temperature presumably because the probe is temperature compensated.

Table 1. Wellhead parameters measured during pumping well test.

| Date/Time | Dissolved Oxygen (mg/L) | Specific Conductance ($\mu\text{S}/\text{cm}$) | Temperature (°C) | pH |
|-----------------------|--------------------------------|--|-------------------------|-----------|
| 8/7/2003 11:10:00 AM | 1.4 | 635 | 18 | 6.70 |
| 8/7/2003 11:30:00 AM | 1.0 | 630 | 17.9 | 6.55 |
| 8/7/2003 11:45:00 AM | 0.95 | 653 | 17.8 | 6.42 |
| 8/7/2003 12:02:00 PM | 0.63 | 665 | 18.5 | 6.48 |
| 8/7/2003 12:50:00 PM | 0.42 | 644 | 22 | 6.57 |
| 8/7/2003 1:50:00 PM | 0.88 | 645 | 21.2 | 6.52 |
| 8/7/2003 2:50:00 PM | 0.80 | 639 | 21.4 | 6.55 |
| 8/8/2003 10:05:00 AM | 0.43 | 645 | 17.4 | 6.95 |
| 8/8/2003 1:20:00 PM | 1.08 | 642 | 22.7 | 6.65 |
| 8/11/2003 9:45:00 AM | 0.52 | 651 | 17.2 | 7.05 |
| 8/11/2003 10:50:00 AM | 0.25 | 648 | 18.4 | 6.81 |

In addition to being a general wellhead parameter, dissolved oxygen is the most thermodynamically favored electron acceptor used by microorganisms for the biodegradation of organic carbon, whether natural or anthropogenic (including some chlorinated aliphatic hydrocarbons [CAHs] of concern at the Building 71b site). Anaerobic bacteria, which favor the reductive dechlorination of CAHs, generally, cannot function at DO concentrations greater than about 1.0 mg/L. The majority of DO concentrations reported in Table 1 are at or below 1.0 mg/L suggesting that reducing conditions probably exist in the water-bearing zone penetrated by well

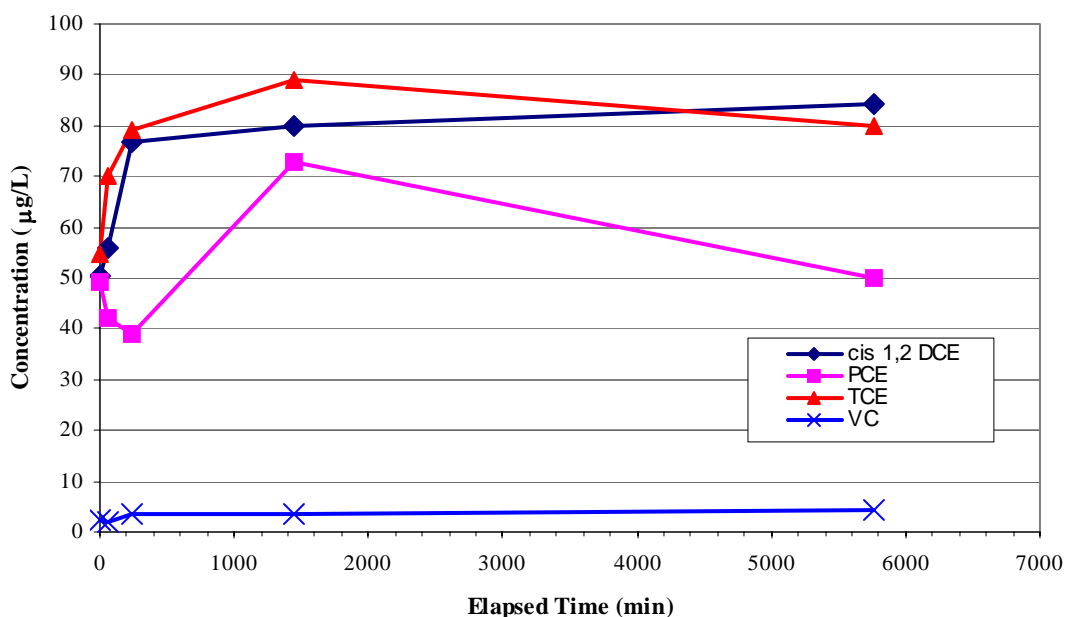
98-13 and that conditions favoring reductive dechlorination of CAHs by anaerobic bacteria potentially exist.

Water Quality Results

Groundwater samples were collected from the discharge water pumped from 98-13 during the test and subsequently analyzed to determine the type and concentration of CAHs present. In addition, samples were taken over time to determine the effect that pumping had on CAH concentrations. Five samples were collected and analyzed for CAHs using USEPA Method 8260B performed by LBNL's Environmental Measurements Laboratory.

Four volatile organic compounds were detected in the five groundwater samples including cis 1,2-dichloroethene (DCE), trichloroethene (TCE), tetrachloroethene (PCE) and vinyl chloride (Figure 8). Detection of these constituents is consistent with previous sample results. The detection of the parent product TCE and PCE and their degradation by products including DCE and VC is encouraging. These results coupled with the low DO values reported earlier suggests that reductive dechlorination by anaerobic bacteria may be taking place or that, at a minimum, conditions potentially exist that favor these processes.

The CAH concentrations were found to increase with time and then stabilize with the exception of PCE, which showed mixed results (Figure 8). A stable or increasing trend in the concentration levels is favored over a decreasing trend if a pumping well is used to enhance the hydraulic gradient and speed the movement of water and dissolved CAHs through the saturated zone during the pilot test. Hydrogen release compounds (HRC®) will be injected into the water-bearing zone



upgradient from the pumping well during the pilot test to enhance biodegradation and decrease the concentration levels of CAH accordingly. Thus, if a decrease in CAHs levels is observed during the pilot test in direct contradiction to the observations made during this pumping well test, then one can safely assume that HRC® enhanced biodegradation is likely taking place. Had the concentration levels decreased during the pumping well test (because of dilution of contaminated water with fresh water drawn in by the well outside the impacted area) then it would be more difficult to draw a definitive conclusion regarding degradation.

Summary

Data collected during this pumping well test were evaluated and interpreted as follows:

- The drawdown data collected during the test were not of sufficient quality (i.e., constant rate and/or constant head conditions did not materialize during the test) or quantity (i.e., duration) to provide an estimate of formation properties using transient well analysis techniques;
- A initial estimate of the hydraulic conductivity (K) equal to 4.0E-07 m/s was determined from the test data using an analytical solution describing steady flow to a well pumped at a constant rate and fully penetrating a confined aquifer. The estimate for K is believed to be too low given the fact that drawdown occurred in a monitoring well located 26 feet from the pumping well and the estimated linear velocity (determined using K) would not have predicted this observation. This suggests that either the steady-state solution is inappropriate for use or the input parameter values, especially the saturated thickness (b), were not adequately defined. Preferential flow paths through the water-bearing layer to the well would reduce b and increase K producing a more realistic value of K;
- Wellhead parameters including dissolved oxygen, temperature, specific conductance and pH stabilized early during the pumping well test suggesting samples collected and analyzed were representative of *in situ* conditions;
- Dissolved oxygen levels measured in groundwater samples collected from the pumping well were at or below 1 mg/L signifying reducing conditions. Anaerobic bacteria that degrade CAHs by reductive dechlorination favor these conditions;
- Groundwater samples collected during the test and analyzed for CAHs contained PCE and TCE that have been shown in the literature to be degraded by reductive dechlorinators;
- With the exception of PCE, the concentration levels of the four CAHs detected in the groundwater samples increased and/or stabilized during 4 days of pumping. An increase in concentration is preferred over a decrease. This is because the introduction of HRC® during the pilot test is expected to enhance biodegradation and reduce contaminant levels. This trend is opposite of the increase observed during this pumping well test and, therefore, will provide credible evidence that the HRC® is working should a decrease in CAH concentrations be observed during the pilot study.

Recommendation

Preliminary analyses and conditions reported herein favor the use of HRC® to enhance biodegradation at the Building 71b well MW71b-98-13 location. The pilot study should proceed to determine if the application of HRC® enhances biodegradation of CAHs at this site.



E.O. Lawrence Berkeley National Laboratory
University of California
Environmental Restoration Program



United States Department of Energy

BUILDING 71B *IN-SITU* CHEMICAL OXIDATION PILOT TEST

for the
Lawrence Berkeley National Laboratory
Environmental Restoration Program

May 2004

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*A Joint Effort of
Environment, Health and Safety Division and
Earth Sciences Division
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May 2004

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This work was done at the Lawrence Berkeley National Laboratory, which is operated by the University of California for the U. S. Department of Energy under contract DE-AC03-76SF00098.

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SECTION 1

SUMMARY

During the weeks of June 16th, 2003, and October 13th, 2003, pilot tests of the in-situ chemical oxidation (ISCO) of chlorinated volatile aliphatic hydrocarbons contaminants (VOCs) consisting primarily of tetrachloroethylene (PCE) in groundwater within the soil and Orinda Formation in the vicinity of Building 71B were performed via injection of citric acid and hydrogen peroxide according to the “Workplan for In-Situ Chemical Oxidation Pilot Test” dated May 2003 (“workplan”). The injection events at Building 71B carried out the pilot test design specified in the workplan to the extent possible given the low permeabilities encountered at the site.

The reagents were injected into the medium and deep intervals during both the June and October injection events. The first event entailed injection into 9 wells and the second into 6 wells. In both events, the wells were arranged in a hexagonal array with a 4 to 5-foot spacing. These arrays enclosed three groundwater wells in the first event and two groundwater wells in the second event.

The injection was performed by Rejuvenate under the supervision of personnel from Lawrence Berkeley National Laboratory’s (LBNL’s) Environmental Restoration Program (ERP) and Parsons Engineering. Concentrations and volumes of reagents injected into each group of wells were recorded along with the injection pressures at each well. The effect of the injection was monitored in real time via measurements of pH in the groundwater wells and observations of the seepage patterns.

Injection pressures were successfully limited to avoid hydraulic fracturing with one exception each in the deep and middle intervals. The pH measurements in the groundwater wells indicate that a radius of influence greater than 2 feet was achieved around the injection wells in the deep interval and less than 2 feet around the wells in the middle interval. This resulted from injection of 90% of the acid mass and 40% of the hydrogen peroxide mass specified in the workplan in the deep injection interval, and 41% of the acid mass and 14% of the hydrogen peroxide mass specified in the workplan in the middle interval. Low permeabilities encountered

in the pilot test area made it unfeasible to inject larger amounts of reagent in an economically-viable time frame.

The reagent solutions injected would occupy a maximum of 14% and 5% of the total pore volume around each well in the deep and middle intervals, respectively, if all of the reagents flowed away from the injection wells via pore flow. However, a significant portion of the reagent volume injected in the deep interval entered a hydraulic fracture and likely advected to positions outside the target pilot test volume. Nonetheless, the injected volume in the deep interval likely occupied a half of the effective pore volume within the target volume. This was sufficient to achieve the desired radius of influence in the more permeable portions of the subsurface.

The VOC concentration changes in response to the two injection events varied with increases in one groundwater well and no change or decreases in the other two in the week following the injection. Total VOC concentration changes were less than a factor of 2. During the two weeks to two months following the injection events, total VOC concentrations returned to near pre-injection levels typically, particularly when examined on a molar rather than a mass basis.

The most significant concentration changes were increases in the concentrations of trichloroethylene (TCE), cis-1,2 dichloroethylene (cis-1,2 DCE), and/or vinyl chloride (VC) to levels above the pre-test concentrations during the one to two months after each injection event. These changes indicate that some reductive dechlorination process was instantiated by the reagent injection. This may be a biotic process using the citrate as a growth substrate, or an abiotic process of some undetermined type. Given these results, a technology to enhance/instantiate reductive dechlorination is probably more likely than ISCO to successfully remediate the site.

SECTION 2

INTRODUCTION

During the weeks of June 16th, 2003, and October 13th, 2003, personnel from Rejuvenate conducted a pilot test of the *in-situ* chemical oxidation (ISCO) of VOCs in the subsurface adjacent to and beneath the southern side of Building 71B as shown in Figure 1. The objectives of this test, as stated in the workplan, were to determine if ISCO could effectively reduce the contaminant mass in soil and groundwater in the vicinity of Building 71B. This work was performed under the direction of personnel from Parsons Engineering and LBNL's ERP.

As described in the workplan, the pilot test location was selected to focus ISCO in the volume beneath the highest VOC concentrations in soil measured at the time. While the majority of these soils were previously excavated during a source removal interim corrective measure (ICM), VOC concentrations in groundwater beneath this excavation, and time variation of these concentrations, suggested that residual contaminants within the zone of water table fluctuation continued to impact groundwater. Therefore the workplan called for injection of reagents into the upper portion of the saturated zone. Soil sampling during the pilot test indicated that VOC concentrations in soil similar to those excavated during the source removal ICM continue to exist beneath the concrete deck to the north of the source ICM area. Therefore the pilot test area was situated at the edge of an area of significant soil contamination in the unsaturated zone as well as at a location of suspected residual contamination in the zone of seasonal saturation.

The volume selected for ISCO contained a mix of soil and underlying Orinda Formation material. No specification to inject iron was made based upon analysis of the iron content of the colluvium at the site. No analysis of the iron content in the Orinda Formation was carried out however. Prior to the conduct of the pilot test, an iron concentration measured in the groundwater from MW71B-99-3R within the pilot test area was considered with the conclusion that groundwater in this formation likely had sufficient iron to allow for ISCO without the addition of iron.

SECTION 3

HYDROGEOLOGIC BACKGROUND

Estimating the average linear velocity of flow in the pilot test area is useful to interpreting the ISCO results. The gradient between wells MW71B-99-3R, and SB71B-03-1 and -2 varied from a low of 0.19 in September, 2003 (the “dry season”) directed to the south to southwest, to a high of 0.26 in February, 2004 (the “wet season”) directed to the southwest. The water table in September, 2003 was located in the Orinda Formation and the water table in February, 2004 was located in colluvium.

The gradient at the pilot test site is approximately perpendicular to the west to northwest strike of the Orinda Formation in the vicinity of the pilot test. The dip of the Orinda Formation in the vicinity of the pilot test is 30 to 40 degrees to the northeast. Geologic logs in the pilot test area indicate the Orinda Formation consists of interbedded siltstone and sandstone as is typical of this formation at LBNL. The relationship of the gradient to the bedding suggests that the bulk hydraulic conductivity should be the harmonic average of the hydraulic conductivities of the individual strata. However, due to the fluvial deposition of the Orinda Formation, the different lithologies occur in a three-dimensional structure which would likely allow the more conductive lithologies to have a greater influence on the bulk conductivity. Therefore the logarithmic average conductivity, which yields a higher estimate than the harmonic average, will be used to estimate the linear velocities in the pilot test area.

The logarithmic average of the hydraulic conductivities inverted from slug test data collected from wells installed in the Orinda Formation throughout LBNL is 1×10^{-7} meters/second (m/s). The log average hydraulic conductivity from wells screened exclusively in fine-grained sandstones and finer-grained rocks is 4×10^{-8} m/, and in wells with some exposure to medium-grained sandstones and coarser-grained rocks is 4×10^{-6} m/s. This latter group includes approximately one quarter of the wells screened in the Orinda Formation at LBNL.

One of the five logged borings in the pilot test area encountered medium-grained sandstone. Lithologies encountered in the other borings were finer grained. Therefore the ratio of

borings at the pilot test area which encountered medium-grained sandstone or coarser lithologies to those borings which did not is approximately the same as that in the entire set of slug-tested wells screened in the Orinda Formation at LBNL. Therefore application of the log average hydraulic conductivity from the entire set of slug test results to the pilot test site is warranted.

The colluvium at the pilot test site consists of clay. The log average hydraulic conductivity from slug tests in wells screened in colluvium at LBNL is 2×10^{-8} m/s, which is one-fifth of the log average hydraulic conductivity of the Orinda Formation. The colluvium at these wells screens consists of clay and gravelly clay.

A review of dry density and moisture content measurements on samples from the Orinda Formation recorded on geotechnical bore logs indicates the porosity of the Orinda Formation rocks averages 25%. Numerical modelling of the groundwater flow in the Old Town area of LBNL indicates that the effective porosity of the Orinda Formation is 3% to 5%. This appears to be a reasonable estimate as flow through any porous rock typically occurs primarily through a fraction of the total pore volume, and in the Orinda Formation flow occurs primarily through the coarser-grained rocks, which make up half or less of the total rock mass.

Using the gradients and hydraulic conductivity from above, an effective porosity of 5%, and assuming groundwater flow follows the hydraulic gradient, the average linear velocity in the pilot test area under background conditions is 3.8×10^{-7} m/s, or 0.11 feet/day (ft/d), and 5.2×10^{-7} m/s, or 0.15 ft/d, at the time of the June and October injection events, respectively.

SECTION 4

JUNE INJECTION EVENT

4.1 SCHEDULE OF ACTIVITIES

On June 12 and 13, 2003, Ofiario Drilling bored and Rejuvenate installed injection wells SB71B-03-R1 to R9. On June 16th, Rejuvenate connected the well heads and injection lines and commenced injection. Injection in the deepest interval continued on June 17th. On June 18th, the injection wells were raised and the middle and deepest intervals were injected. This work was conducted under the supervision of Parsons Engineering.

4.2 INJECTION WELLS

The June injection was conducted in 9 wells consisting of SB71B-03-R1 through R9. These wells were arranged in a nearly hexagonal grid with a 4 to 5-foot spacing, and surrounded three groundwater wells as shown on Figure 1. The injection wells were installed to depths greater than well screens in SB71B-03-1 and -2 and a depth overlapping with the upper portion of the screened interval in MW71B-99-3R.

The injection wells consisted of ¾-inch internal-diameter, stainless-steel pipe with external threaded couplings. The screen sections consisted of the same pipe with three, approximately 3/16-inch holes drilled at equal angles around the pipe. A set of these holes was drilled at approximately 4-inch intervals along the screen. Chemical resistant rubber packers could variously be attached above the screen or above and below the screen. The outer diameter of the packers was approximately 1.5 inches when deflated. The packers were wrapped around the injection casing and designed to be inflated by the injectate itself via holes drilled through the casing. A typical well configuration is shown on Figure 2.

The injection wells were installed in 3.5-inch diameter open borings advanced to 26 feet below ground surface (bgs) by a portable hydraulic drill rig using continuous flight augers. A 2-inch internal diameter PVC casing was grouted into the upper few feet of each boring. The

annulus between the PVC casing and the injection casing was sealed at the top of the PVC casing by a compression fitting in order to minimize exposure to the injectate if a packer failed in the boring during injection.

4.3 INJECTION INTERVALS

On June 16th and 17th, the packers were placed to restrict injection to the interval below 18 feet. On June 18th, the injection wells were raised to restrict injection to the interval below 10 feet.

4.4 INJECTION PRESSURES

Injection pressures were generally 10 psi or less during injection of the both the deep interval and the middle and deep interval. The injection pressure was selected by Rejuvenate.

4.5 REAGENT QUANTITIES INJECTED AND REAL-TIME RESPONSES OBSERVED

Approximately 1,060 gallons of reagent were injected. This consisted of 740 gallons of 11% hydrogen peroxide solution, on average, and 320 gallons of 13% citric acid, on average. Approximately 650 gallons were injected into the deep interval and 410 gallons into the middle and deep interval over the course of three days as listed in Table 1. The total elapsed injection time to the deep interval was 2 hours and 25 minutes, and to the middle and deep interval was 1 hour and 5 minutes. Therefore the average injection rate to the deep interval was 4.5 gallons per minute (gpm), or 0.65 gpm per well, and to the middle and deep interval was 6.3 gpm, or 0.9 gpm per well.

Table 1. Injection Periods During the June Injection Event.

| injection | | | SB71B-03 wells injected during period |
|------------------|----------------|-----------------|--|
| No. | period | interval | |
| 1 | 6/16 afternoon | deep | R2, R3, R4, R5, R6, R7, R9 |
| 2 | 6/17 morning | deep | R2, R3, R4, R5, R6, R7, R9 |
| 3 | 6/17 afternoon | deep | R1, R2, R3, R4, R5, R6, R8 |
| 4 | 6/18 morning | middle and deep | R2, R3, R4, R5, R6, R7, R9 |
| 5 | 6/18 afternoon | middle and deep | R2, R3, R4, R5, R6, R7, R9 |

Approximately 650 gallons were vacuumed into drums as seepage from the ground surface. The seepage was concentrated at the margins of a low-strength concrete backfill through which several of the injection wells were installed. The backfill was placed in an approximately 10 foot deep source removal excavation in 2000. The pH of the seepage was 3 to 4. Some small quantity of seepage (approximately 20 gallons) in addition to this amount ran down a slope away from the site and may have entered a storm drain catch basin. Monitoring of flow in a nearby downstream catch basin shortly after this event showed neutral pH.

Table 2 lists pH measurements taken from MW71B-99-3R and SB71B-03-1 after some of the injection periods. SB71B-03-2 was dry prior to injection and remained so during injection.

Table 2. Summary of pH Measurements During the June Injection Period.

| injection period | | | well | |
|------------------|----------------|-----------------|-------------|------------|
| | | | MW71B-99-3R | SB71B-03-1 |
| No. | time | interval | after | after |
| 1 | 6/16 afternoon | deep | | |
| 2 | 6/17 morning | deep | | 6.0 |
| 3 | 6/17 afternoon | deep | 7.0 | 6.0 |
| 4 | 6/18 morning | middle and deep | | 6.0 |
| 5 | 6/18 afternoon | middle and deep | | |

4.6 PACKER FAILURE

The high ratio of the seepage volume (>670 gallons) to the injectate volume (approximately 1060 gallons), and the spatial distribution of the seepage led to the hypothesis that Rejuvenate's packer system was failing to seal properly against the borehole walls. Such a failure would allow the injectate to occupy the entire well bore and would not be noticed at the ground surface due to the compression fitting at the top of each well. Having occupied the entire well bore, the injectate would enter the least resistive location along the entire borehole wall. It was hypothesized that this location would be the interface between the low-strength concrete backfill and the underlying *in-situ* material.

Review of Rejuvenate's packer system, which had previously been utilized at a number of other non-LBNL sites under the supervision of other consultants, revealed that the inflation of the packers by the pressurized injectate inside the well casing would not be sufficient to inflate

the packers against the borehole wall under low flow conditions, such as are likely at the 71B pilot test site due to the low permeabilities of the surrounding hydrogeologic materials. Under low flow conditions, pressures outside the injection screen would tend to match pressures inside the screen due to little head loss across the screen. Due to the material characteristics of the packers, they require pressure inside the packer to exceed the pressure outside the packer by approximately 15 pounds per square inch (psi) to begin inflation. Therefore the packers would not inflate under low-flow conditions.

Based upon this review, Rejuvenate agreed to fabricate packers with a separate pressure line and reinject the site. This was carried out during the October injection event reported on below. The difference in flow rates between the June injection event with the original packer system, and the October injection event with the independently pressurized packers provides further evidence of the failure of Rejuvenate's packer system to properly seal. The June flow rates were 0.65 gpm into a supposed 8-foot long interval and 0.9 gpm into a supposed 16-foot long interval, both at 10 psi. The October flow rates were 0.15 gpm at 10 psi and 0.35 gpm at 20 psi into 5-foot long intervals. If the original packer system had sealed properly, this would imply the October flow rates should have been 0.3 to 0.4 gpm at 10 psi and 0.6 to 0.8 gpm at 20 psi into 5-foot long intervals, or approximately twice the actual flow rates achieved in the October injection event.

After the review of the packer system, Rejuvenate contended that their packer system worked as designed in more permeable settings which allowed higher flow rates. However as the system is based fundamentally upon dynamic pressures developed during injection, there is significant uncertainty whether the head loss across the screen in high-flow conditions would be enough to produce the pressure differential required for this packer system to seal the borehole. Rejuvenate's claim would have to be proven on a site by site basis, perhaps by installing pressure instrumentation outside the injection screen, in order to assure that the injectate pushed in at high flow rates was actually going into the intended formation interval.

4.7 DISCUSSION OF RESULTS

The workplan called for injection of 7.3 kg of citric acid in 9 gallons of solution and 37 kg of hydrogen peroxide in 97 gallons of solution per 5-foot interval for injection wells spaced 5 feet apart. Assuming that the quantity of injectate which was not collected as seepage remained in the subsurface, the average quantities injected in each approximately 22-foot long (the entire well depth less the average thickness of concrete) interval were 7.0 kg of citric acid and 12.2 kg of hydrogen peroxide in 46 gallons of solution. Taking the well spacing as 4.5 feet, 27% of the acid mass, 9% of the hydrogen peroxide mass, and 14% of the total reagent volume specified in the workplan were injected throughout the entire well depth. The pH measurements during the injection event indicate that the maximum radius of influence from the injection wells was less than the typical two-foot minimum distance from an injection to a monitoring well.

The VOCs repeatedly detected in the three wells within the pilot test area consist of PCE, TCE, cis-1,2 DCE, and VC. Of the three wells enclosed in the pilot test area, MW71B-99-3R has the longest concentration history prior to the pilot test. One baseline sample was collected from SB71B-03-1 prior to the test and SB71B-03-2 was dry prior to the test.

The VOC concentrations in MW71B-99-3R during the month after the June injection event were similar to concentrations at this time of year prior to the injection event as shown on Figures 3 and 4. This indicates that on the timescale of the activity of hydrogen peroxide, typically believed to be hours in the subsurface, no significant oxidation of the contaminant mass occurred at the position of this well. Two months after the injection event, the total concentration of VOCs on a mass basis decreased slightly and the VOC ratios changed significantly. The fraction of PCE decreased, and the fraction of cis-1,2 DCE, and to a lesser extent TCE, increased to ratios which have not been measured in this well previously.

The month time-scale of the change in VOC ratios in MW71B-99-3R indicates that a process other than chemical oxidation was induced by the injection. Based upon the change in VOC ratios, it appears that a biotic or abiotic reductive dechlorination process occurred. A biotic process may have been triggered by the presence of citrate, which is an organic compound which potentially could have been utilized as a growth substrate.

No significant changes clearly attributable to the injection of chemical oxidants are observed in the VOC concentrations in SB71B-03-1 as shown on Figure 5. The concentration of PCE immediately after the injection was less than half that prior to the injection, however the concentration of the other VOCs remained approximately constant. After the initial decline following the June injection event, the PCE concentration remained relatively constant for more than two months after the June injection event. From the average linear velocity of 0.11 ft/day, the average distance of groundwater advection during the two months following the June injection event is approximately 7 feet under ambient conditions. As SB71B-03-1 was at the upgradient edge of the injection grid, and the radius of influence was apparently less than 2 feet, the initial PCE concentration decrease and following stabilization is therefore probably due to well equilibration.

After the injection, groundwater was present henceforth in SB71B-03-2. The initial PCE concentration of approximately 900 ug/L after injection decreased to approximately 200 ug/L during the following month and stabilized at this concentration. This is likely due to mobilization of contaminants residing in the vadose zone just north of the well as discussed below in the October injection results section. From the average linear velocity estimate, the average groundwater advection distance following injection is approximately 3 feet. As SB71B-03-2 is also at the upgradient edge of the injection grid, the decrease in concentrations is probably due to advection of injected water contaminated by residual contaminants in the vadose zone away from the well, and advection of relatively cleaner groundwater from upgradient of the injection zone of influence to the well.

SECTION 5

OCTOBER INJECTION EVENT

5.1 SCHEDULE OF ACTIVITIES

On September 16th, Ofiario Drilling bored and sampled SB71B-03-R10 and R11. Also on this date, injection wells SB71B-03-R1 to R2 were removed and the associated borings examined with a downhole video camera to determine their suitability for reuse in the second injection period. This examination revealed that the borings were filled with slough below 13 feet bgs and therefore were inappropriate for reuse. Therefore Ofiario Drilling overdrilled SB71B-03-R1 to R4 and subsequently sealed the borings with bentonite chip on September 16th and 18th to prevent vertical migration of injectants during the second injection period. Replacement borings SB71B-03-R12 to R15 were drilled by Ofiario Drilling on September 18th.

Installation of the injection wells in SB71B-03-R10 to R15 by Rejuvenate on October 7th failed due to significant leakage between the packer line and injection line inside the well heads. The injection well heads were retooled at Rejuvenate's shop and the injection wells were installed into the borings SB71B-03-R10 to R15 on October 14th. The well pattern utilized for the October injection event consisted of SB71B-03-R12 to R15 which were arranged 4 to 5 feet apart in a nearly hexagonal grid enclosing two observation wells, SB71B-03-1 and SB71B-03-2, as shown on Figure 1. Injection in the deep interval occurred on October 15th. On October 16th the injection wells were removed, reconfigured for injection in the middle interval, and reinstalled. The middle interval was injected on October 17th. This work was conducted under the supervision of LBNL's ERP and Parsons Engineering.

5.2 INJECTION WELLS

The October injection was conducted in 6 wells consisting of SB71B-03-R10 to R15. These wells injection wells were arranged in a nearly hexagonal grid with a 4 to 5-foot spacing, and surrounded two groundwater wells as shown on Figure 1. The injection wells were installed to similar depths greater than groundwater wells SB71B-03-1 and -2

The packers utilized during the October injection event utilized packers fabricated by Rejuvenate which were inflated with water independent from the injectate line. The outside diameter of these packers was approximately 2.5 inches. Due to the size of these packers, the near-surface PVC casings installed prior to the June event were not installed in the borings prior to the October event. In other respects the wells were the same as those used in the June injection.

The wells were again installed in 3.5-inch diameter open borings. Borings SB71B-03-R10 and R11 were advanced to 27 feet bgs, while borings SB71B-03-R12 to R15 were advanced to 25 feet bgs. This difference accounted for the difference in ground surface elevation between the borings such that the bases of the borings were at approximately the same elevation.

5.3 INJECTION INTERVALS

Table 3 below lists the injection intervals in each well along with the estimated depth to the base of concrete and top of the Orinda Formation at each well.

Table 3. Geologic Contacts and Injection Intervals.

| well name | depths ft) | | | | | |
|--------------|------------------|-------------------------|----------------|---------------|-----------------|-------------------------------|
| | base of concrete | top of Orinda Formation | bottom of hole | deep interval | middle interval | shallow interval [†] |
| SB71B-03-R10 | 1.5 | 9.5 | 27 | 22-27 | 11-17* | 4-10 |
| SB71B-03-R11 | 1.5 | 17 | 27 | 22-27 | 11-19 | 4-10 |
| SB71B-03-R12 | 2.5 | 5 | 25 | 20-25 | 14-19 | 7-12 |
| SB71B-03-R13 | 8 | 9 | 25 | 20-25 | 13-18 | 9-14 |
| SB71B-03-R14 | 4 | 13 | 25 | 20-25 | 12-17 | 6-11 |
| SB71B-03-R15 | 2 | 16 | 25 | 20-25 | 12-17 | 6-11 |

*not injected due to packer failure, [†] not injected due to time constraints

Due to concerns about the inability of the packers to seal against a previously injected borehole wall, the depths of the each interval were selected such that the top of the packer below

the screen would inflate against an uninjected section of borehole wall. This decision resulted in an uninjected interval ranging from 1 to 3 feet in length between the intervals. The top of the shallowest interval was selected to minimize interaction of the injectate with the overlying concrete structural slab at R10 and R11 and low-strength backfill at the remainder of the wells). Within these constraints, the boundary between the shallow and middle injection intervals was selected to minimize exposure of more than one hydrogeologic unit within an interval.

Due to the low injection rates, the time scheduled for injection did not allow for injection of all three intervals. Therefore a decision was made not to inject the shallow interval as it was in the unsaturated zone, and the metric of success for this pilot test, according to the workplan, was the concentration of VOCs in groundwater. The middle interval in SB71B-03-R10 could not be injected due to failure of the lower packer during reinstallation of the well. The failure of this packer led to the introduction of 10s of gallons of water into this boring. Water was observed exiting the top of the boring following both attempts to set this well at the middle injection interval and inflate the well.

5.4 INJECTION PRESSURES

Based upon the instability of the hydrogen peroxide, chemical oxidation using this reagent is generally believed to be effective for only a matter of hours after injection. Therefore, advection driven by injection pressure must bring the reagents into contact with the target contaminants to be oxidized within a very short time period. This is unlike more persistent *in-situ* treatment technologies where natural groundwater advection may be relied upon to bring the reagents, or their byproducts, into contact with the target molecules. The above suggests that injection pressures must be controlled carefully to prevent hydraulic fracturing of the formation that would cause the reagents to bypass most of the pore volume containing the target contaminants.

Hydraulic fracturing from vertical wells in the shallow subsurface is generally related the vertical stress in the material around the well, which is typically equal to the overburden pressure. The minimum overburden pressure in each injection interval occurs at the shallowest portion of the interval. Review of dry density and moisture content measurements for engineered fill, colluvium, and the Orinda Formation at other locations at LBNL indicates a total density of 120 pounds per cubic foot (lbs/ft³) is a conservative approximation for the average

total density above the water table at the 71B pilot test site. This value equates to a minimum overburden pressure of 17 psi in the deep injection interval and 9 psi in the middle interval. The maximum injection pressures were set to slightly above these values to account for the higher than overburden pressures typically required to initiate a hydraulic fracture. Therefore the maximum target injection pressure for the deep interval was 20 psi and for the shallow interval was 10 psi.

Significant expansion of the packers occurs at approximately 15 psi. This conclusion was based upon discussions with Rejuvenate as well as direct observation of partial inflations of packers laid out on the ground surface. This suggests only the increment of packer pressure above 15 psi is effective at resisting passage of injected reagents. During injection, the target packer pressure was set at 15 psi higher than the maximum target injection pressures, or approximately 35 psi for the deep interval and 25 psi for the shallow interval. With exceptions detailed below, the injection pressures and packer pressures were maintained according to the specifications described above.

5.5 INJECTION INTERVAL PERMEABILITY

The permeability of each interval in each well was qualitatively gauged by injecting acid at equal to or less than the maximum injection pressure for fifteen minutes or until approximately 4 gallons of acid had been injected. The first 2 gallons injected were sufficient to fill the 5 foot-long boring interval with reagent. Fifteen minutes to inject an additional 2 gallons into the formation (equivalent to 0.15 gpm) was selected as it is a lower bound for the economic feasibility of *in-situ* treatment via reagent injection. At this rate, approximately 70% of the reagent volume specified in the workplan could be injected in an 8-hour period.

Injection pressures at the truck manifold and the well head provided secondary confirmation of the permeability around well. A well head pressure less than or equal to the manifold pressure on the same injection line indicated flow to the well (the manifold pressure gauges were typically positioned approximately 14 feet above the well head pressure gauges). A well head pressure less than the maximum injection pressure was also taken as indicating flow to the well. Relative differences in the flow rate to each well during multiwell injection, as measured qualitatively by flow

meters on the injection manifold, provided a tertiary confirmation of differences in the permeability around each well.

Based upon data accumulated via the three methods outlined above, approximately half of the injection intervals were sufficiently permeable to accept 2 gallons of reagent within 15 minutes. The deep intervals in SB71B-03-R11, R12, and -R14 passed this test, and the middle interval in SB71B-03-R11 and -R15 passed this test. There was no discernible pattern or cause for which deep injection intervals passed the test. The middle injection intervals which passed the test had the greatest exposure of soil above the Orinda Formation suggesting that the soil at the site is relatively more permeable than the underlying rock, which is the reverse of the application of site-wide slug test results to this site as discussed in Section 2: Hydrogeologic Background.

5.6 REAGENT QUANTITIES INJECTED AND REAL-TIME RESPONSES OBSERVED

Approximately 256 gallons of reagent were injected. No seepage from the ground surface, the top of the injection wells (no compression fitting was present on the wells during this event), or surrounding injection wells was observed during injection, except as described below. These observations confirmed that the independently pressure-controlled packers, in combination with the specified pressure differential, were successful at restricting injection to the intended interval.

The reagents injected consisted of 102 gallons of 12% citric acid, on average, and 154 gallons of 17% hydrogen peroxide solution, on average. Of these totals, 70 gallons of citric acid and 115 gallons of hydrogen peroxide were injected in the deep interval and the remainder was injected in the middle interval. The total elapsed injection times for the deep and middle interval were 3 hours and 2 hours 35 minutes, respectively. Therefore the average injection rate to three wells at a time (the maximum number injected at once) in the deep interval was 1 gpm, or 0.35 gpm per well. The average injection rate to three wells at a time in the middle interval was 0.45 gpm, or 0.15 gpm per well.

During deep interval injection of hydrogen peroxide, the pressure in SB71B-03-R14 spiked, apparently due to offgassing from reactions in the boring. Following this pressure spike, the pressure dropped below pre-spike levels, the flow rate increased, turbid water was observed

in SB71B-03-1, and seepage and offgassing were observed at the ground surface and from SB71B-03-R7 and –R8 to the south. All of these observations are consistent with the development of a hydraulic fracture. Injection to this well was subsequently shut off. Largely as a result of the hydraulic fracture, the majority of the reagents injected in the deep interval were injected in the SB71B-R11, R14 and R15 triangle of wells.

Due to the low injection rate in the middle interval of SB71B-03-R12 and R13, it was decided to initiate hydraulic fractures from SB71B-03-R12 toward the end of the injection event in order to maximize the opportunity for reagents to enter the formation. The injection pressure was increased to 20 psi and subsequent observations indicated a fracture was initiated.

Table 4 lists pH measurements taken from MW71B-99-3R and SB71B-03-1 before, during and after some of the injection periods.

Table 4. Summary of pH Measurements During the October Injection Period.

| injection period | | | well | | | | | | |
|------------------|-----------------|----------|-------------|-------|------------|---------------|-------|------------|-------|
| | | | MW71B-99-3R | | SB71B-03-1 | | | SB71B-03-2 | |
| No. | time | interval | before | after | before | during | after | before | after |
| 1 | 10/15 morning | deep | 7.0 | | 7.0 | | | 7.0 | 4.5 |
| 2 | 10/15 afternoon | deep | | | | 5.5, 4.5, 6.0 | | | |
| 3 | 10/17 afternoon | middle | 6.0 | 6.0 | 6.0 | | 6.0 | 7.0 | 7.0 |

5.7 DISCUSSION OF RESULTS

The workplan called for injection of 7.3 kg of citric acid in 9 gallons of solution and 37 kg of hydrogen peroxide in 97 gallons of solution per 5-foot interval for injection wells spaced 5 feet apart. The average quantities injected in each, 5-foot long deep interval were 5.3 kg of citric acid in 12 gallons of solution and 12.3 kg of hydrogen peroxide in 19 gallons of solution. Taking the well spacing as 4.5 feet, 90% of the acid mass, 40% of the hydrogen peroxide mass, and 36% of the volume specified in the workplan were injected. The average quantities injected in each, 5-foot long (on average) middle interval were 2.4 kg of citric acid in 6 gallons of solution and 4.2 kg of hydrogen peroxide in 8 gallons of solution. Taking the well spacing as 4.5 feet, 41% of the acid mass, 14% of the hydrogen peroxide mass, and 16% of the total volume specified in the workplan were injected. As previously described, however, the distribution of these reagents is

highly non-uniform due to differences in permeability between the wells, and hydraulic fracturing of the interval in some wells. These totals do not account for losses due to seepage, which comprised only a small fraction of the total volume of reagents injected during the October injection event.

The pH measurements during the injection event indicate that the maximum radius of influence from the injection wells was more than the two-foot minimum distance from an injection to a monitoring well during the deep interval injection, and less than the two-foot minimum distance from an injection to a monitoring well during the middle interval injection. A small reduction in pH in MW71B-99-3R from prior to after injection indicates that reagents reached this well suggesting an overall radius of injection influence of greater than 6 feet. However, seepage from injection wells SB71B-03-R7 and R8 during the hydraulic fracturing of SB71B-03-R14 indicates reagents probably reached MW71B-99-3R through the hydraulic fracture.

Analysis of dry densities of engineered fill, colluvium, and the Orinda Formation from other sites at LBNL indicates the porosity of the soils as approximately 38% and of the Orinda Formation as 25%. Based upon this porosity, the void space in the 2.5-foot radius cylinder around each 5-foot long injection interval in the soil and the Orinda Formation is 26 and 37 cubic feet for the deep and middle intervals, respectively. Therefore the reagent solution volumes injected are 14% and 5% of the total pore volumes for the deep and middle intervals, respectively. Note that due to hydraulic fracturing, some of the reagents did not flow away from the injection wells via pore flow, however, and so the actual fraction of the pore volume occupied by reagents within the target volume is somewhat less. Nonetheless comparison of the reagent volumes to the pH observations suggest the radius of influence was due to flow through pathways which were more permeable than the average, and which occupied perhaps a half of the total volume if the effective porosity is taken as approximately a quarter of the total porosity.

The hydraulic heterogeneity at the site indicated above by comparison of the pH responses to the reagent volumes injected is further confirmed by consideration of the injection flow rates. Half of the injection intervals were unable to accept a flow rate of 0.15 gpm or greater during the single well injections, yet the average flow rates were 0.35 gpm and 0.15 gpm per well in the deep and middle interval, respectively. Therefore the variation in flow rate to the

deep interval must have been at least a factor of 3 from the minimum flow rate to the maximum flow rate.

The total VOC concentrations in MW71B-99-3R decreased significantly the day after the pilot test as shown in Figure 4, and the concentrations of individual VOCs decreased in proportion to the total decrease. The total concentration rebounded significantly in the following sample collected three days later. If the total concentration decline had been due to oxidation of a significant portion of the contaminant mass between the injection wells and this monitoring well, the reduction should have persisted for a length of time suggested by the linear velocity and the flow direction. As the injection wells were 6 to 10 feet upgradient from MW71B-99-3R, the post-injection average linear velocity of 0.15 ft/day suggests the concentration reductions due to oxidation should have persisted for a month or more. The much shorter duration of the concentration decrease indicates the decrease is more likely indicative of dilution of groundwater in the well by reagents flowing along a preferential flow path. This path probably consists of the hydraulic fracture which developed during injection in the deep interval in SB71B-03-R14 and propagated into the region around MW71B-99-3R as evidenced by offgassing and seepage at SB71B-03-R7 and -R8 to either side of the monitoring well during injection. Surprisingly, a similar total concentration decrease due to dilution by reagent intrusion was not observed in SB71B-03-1 after hydraulic fracturing at SB71B-03-R14 despite a turbidity spike in this well. This may be because the sample was taken after completion of all injection activities, including the injection of the middle interval near this well.

During the week after the injection, the concentration of TCE and cis-1,2 DCE rebounded to the upper limit of the pre-injection concentration range and the concentration of PCE remained below pre-injection concentrations in MW71B-99-3R. In the month to month and a half after the injection, the concentrations of cis-1,2 DCE and VC increased to levels in excess of pre-injection concentrations, the concentration of TCE decreased to the lower limit of the pre-injection concentration range, and the concentration of PCE decreased further. On a mass basis (milligrams/litre), the total VOC concentration during this period decreased by approximately one quarter as compared to the total concentrations during the same month in previous years as shown on Figure 4. However, the total concentration on a molar basis (mols/liter) remained the

same or slightly above previous years, as shown on Figure 7, indicating that none of the contaminant mass was completely oxidized.

The total VOC concentration in SB71B-03-1 decreased by approximately 40% in the week after the injection as shown on Figure 5. The concentration of each individual VOC decreased as well with the concentration of cis-1,2 DCE decreasing the most and the concentration of PCE decreasing the least. Within two to three weeks after the injection, the total VOC concentration rebounded to 90% of pre-injection levels with the ratio of cis-1,2 DCE to PCE significantly increased. Within four weeks, the total VOC concentration rebounded fully. Significant precipitation did not commence until mid-December, so using the dry season average linear velocity of 0.11 ft/d, the total average advection distance under ambient conditions would have been 2.3 feet in the three weeks after the injection. The actual advection distance would have been somewhat less due to dissipation of the injection pressures. As SB71B-03-1 is centered on the downgradient side of an injection well triangle approximately 4 feet across, and there was some radius of influence around this triangle, the time to rebound is significantly shorter than would be commensurate with uniform oxidation of the contaminant within the cylindrical volume around each injection well. Therefore the time to rebound likely reflects advection and diffusion of untreated groundwater from lower permeability zones not accessed by reagents within the treatment area.

The total VOC concentration in SB71B-03-1 increased by more than 50% in the week after the injection with relative increases in PCE and TCE as shown in Figure 6. This was likely due to mobilization of contaminant from the area north of the well where high concentrations of VOCs were measured in the soil above 10 feet bgs in SB71B-03-R10 prior to the ISCO test. Contaminants from this depth were probably mobilized by water introduced into this boring due to packer failures. Within two months after the injection the total VOC concentration declined to 25% greater than pre-injection levels with a significant increase in cis-1,2 DCE and TCE relative to PCE.

Groundwater from all of the sites had increases in the concentration of cis-1,2 DCE, and MW71B-99-3R also had an increase in VC, indicating again that injection of the chemical oxidation reagents likely initiated a reductive dechlorination process.

SECTION 6

CONCLUSIONS

The workplan was followed to the extent practicable and economical given the low permeabilities encountered at the Building 71B pilot test site. No concentration changes measured in the three wells monitoring the June injection event are apparently due to chemical oxidation. The concentration changes in SB71B-03-1 after the October injection event are apparently due to chemical oxidation. The magnitude of the VOC concentration decrease (40%) and the time to full concentration rebound relative to that predicted under ambient groundwater flow conditions (0.5 month versus 2 to 3 months) indicates the injection was not able to overcome the significant heterogeneity which exists in the Orinda Formation. The concentration increase in SB71B-03-2 after the October injection event is apparently due to mobilization of VOC contaminants residing in the vadose zone to the north.

Based upon these results, full-scale ISCO implemented with the technology used for the pilot test would likely not be successful at permanently lowering VOC concentrations to the likely regulatory limits for the LBNL site. It is likely that ISCO implemented using a technology which allowed for narrower injection intervals, such as sleeve-port injection, would overcome the heterogeneity at the site. This would be predicated upon remediation of the contaminants in the vadose zone to the north of the pilot test area. Remediation of this zone could be by excavation, or some in-situ technology such as chemical oxidation. The pilot test reported on in this document does not provide any data regarding the effectiveness of ISCO to remediate contaminants in the vadose zone.

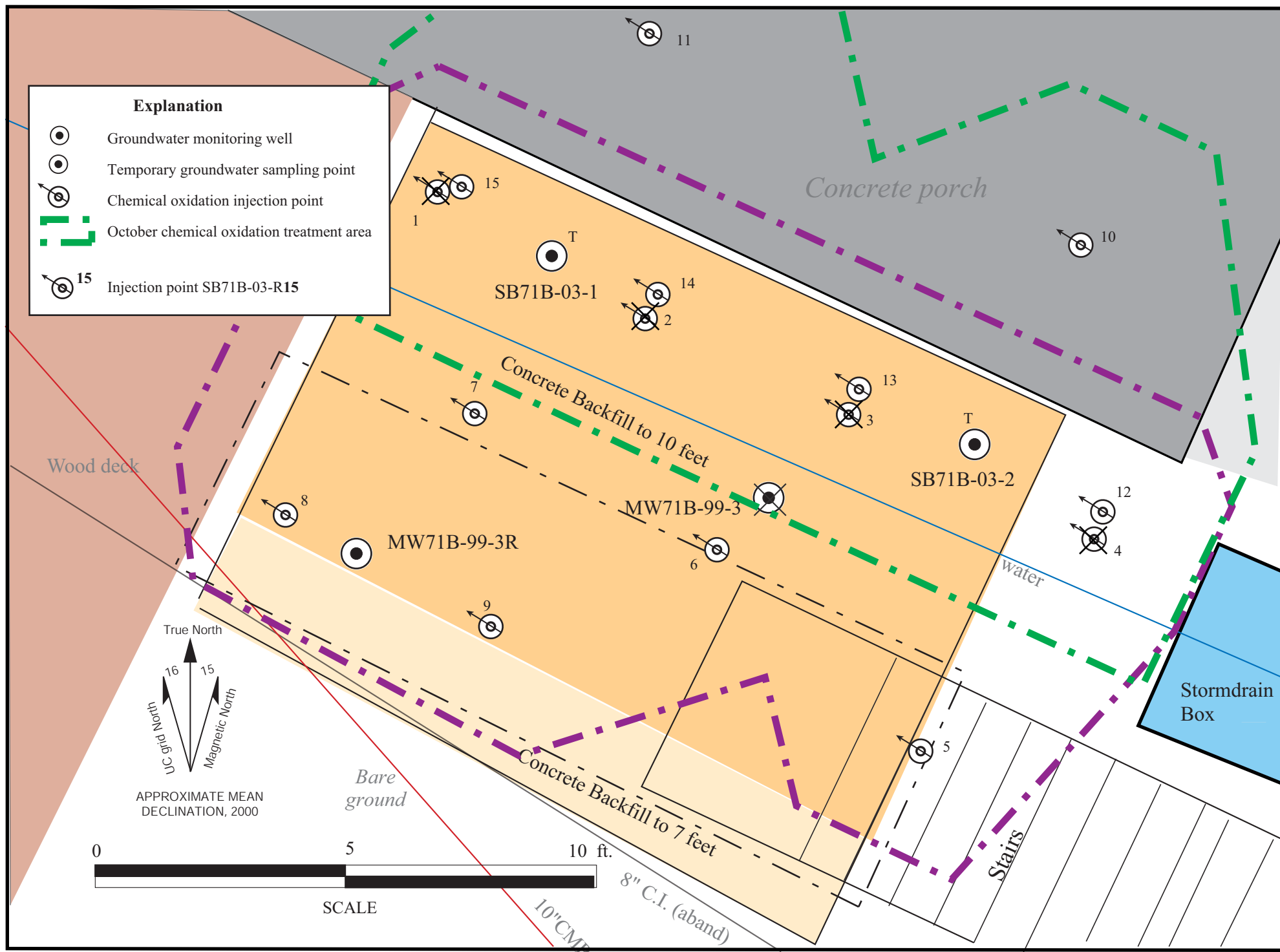
The observed concentration changes (increased concentrations of less chlorinated relative to more chlorinated compounds) suggest that ISCO as implemented in this pilot test fomented reductive dechlorination. The results from MW71B-99-3R indicate this most clearly as shown on Figure 7 by the decline in the average number of chlorine atoms per VOC molecule from nearly 4 (PCE dominant) prior to the June injection event to 2 (DCE dominant) a month after the October injection event.

One hypothesis is that the injected citrate was utilized as a growth substrate by the endogenous microbial community. However, this is an unexpected result as the degradation of hydrogen peroxide would possibly sterilize the injection area as well as elevate the dissolved oxygen concentrations in the subsurface, neither of which is conducive to reductive chlorination. Unfortunately, measurements of dissolved oxygen and the concentrations of different dissolved iron and manganese cations are not available to further analyze this hypothesis.

The apparent occurrence of reductive dechlorination following the ISCO injection suggests that a remedial technique designed to enhance reductive dechlorination would be a successful alternative to ISCO at the subject site.

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10-71B ISCO map test2.ai
10/03

Figure 1 . Locations of in-situ chemical oxidation injection points (Sept. 2003) and temporary groundwater sampling points SB71B-03-1 and SB71B-03-2.


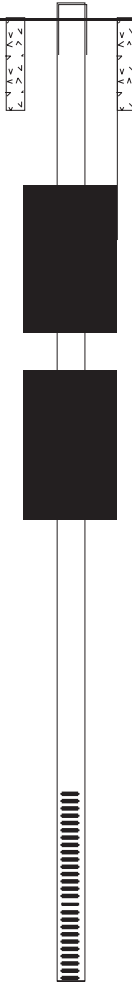
| | | | | | | | | | | | | | |
|---|--|-----------------------------|--|---------------------------|--|---|---------------------------------|---|------------|--|--|----------------|--|
| | | | | SITE: SB71B-03-R1 | | UC EAST: 2630.00 | | UC NORTH: 1185.00 | | UC SURF. ELEV.: 839.00' | | | |
| | | | | LOCATION: South of 71B | | | | | | | | | |
| DATE(S) INSTALLED: 06/12/03 - 06/12/03 | | BORING DIAMETER: 3.50in | | TOTAL DEPTH: 26.50' | | COMPLETED DEPTH: 26.00' | | REMARKS: Well construction depths are approximate. Well removed and boring redrilled to 30 feet below ground surface and backfilled with medium bentonite chip on 9/16/2003. | | | | | |
| DRILLING METHOD: Hydraulic portable with solid-stem auger | | | | LOGGED BY: HP | | | | | | | | | |
| DRILLING CONTRACTOR: Ofaro | | CONSULTING FIRM: Parsons | | | | | | | | | | | |
| GEOLOGICAL MATERIAL DESCRIPTION | | | | | | GRAPHIC LOG | DEPTH (ft) | RECOVERY | BLOW COUNT | | | WELL MATERIALS | |
| CONCRETE, low strength, Not logged below base of concrete. | | | | | |  | 5 10 15 20 25 30 | | |  | grout outside conductor | | |
| | | | | | | | | | | | 2" ID, Schedule 40 PVC conductor packer 3/4" ID, stainless steel casing packer 3/4" ID, ~0.1" perforated screen silt trap | | |
| ENVIRONMENTAL RESTORATION PROGRAM | | | | | | | | REVIEWED BY: | | Page 1 of 1 | | | |

Figure 2. Typical injection well log .

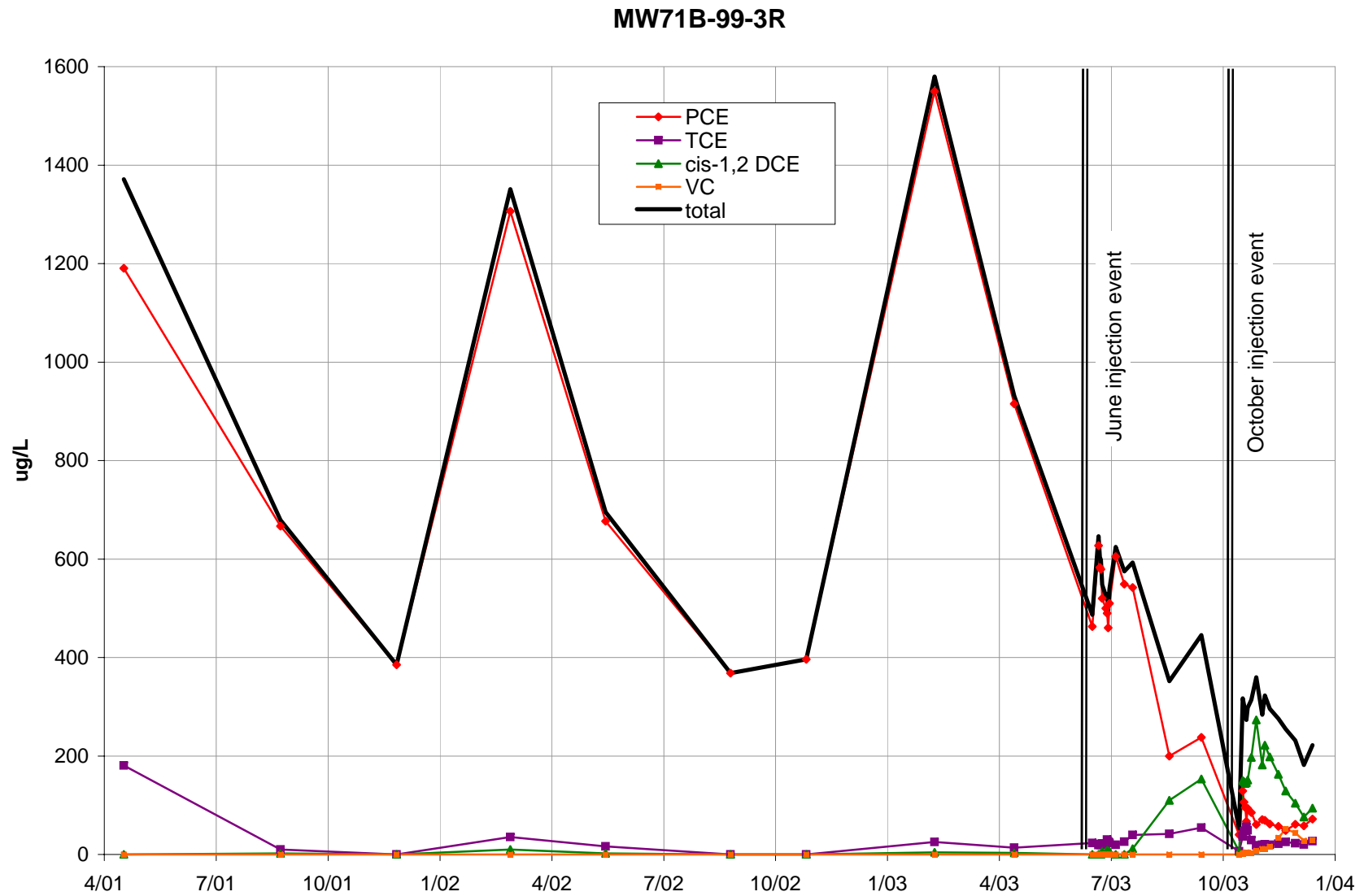


Figure 3. Long-term VOC concentration changes in MW71B-99-3R.

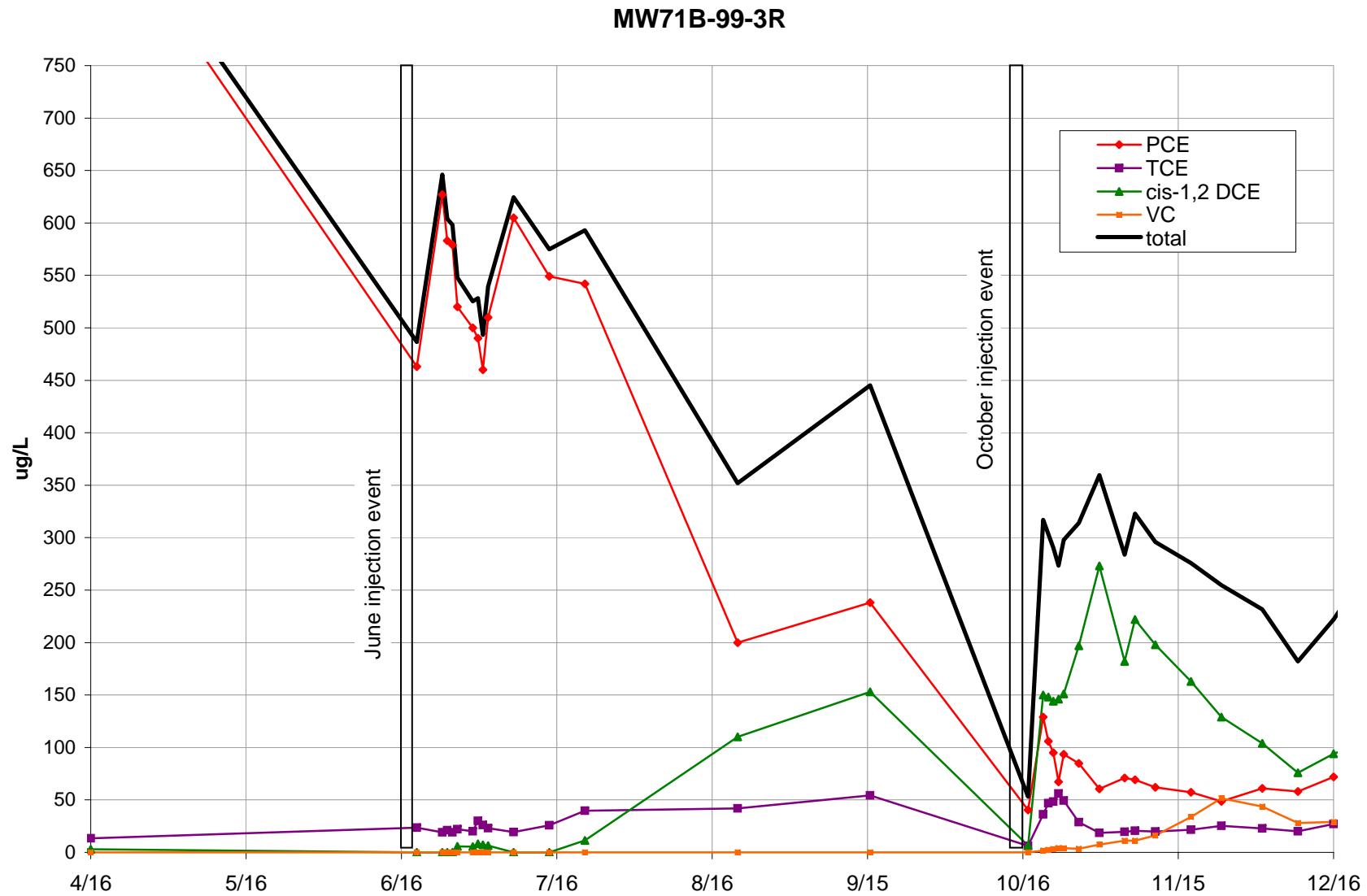


Figure 4. Short-term VOC concentration changes in MW71B-99-3R.

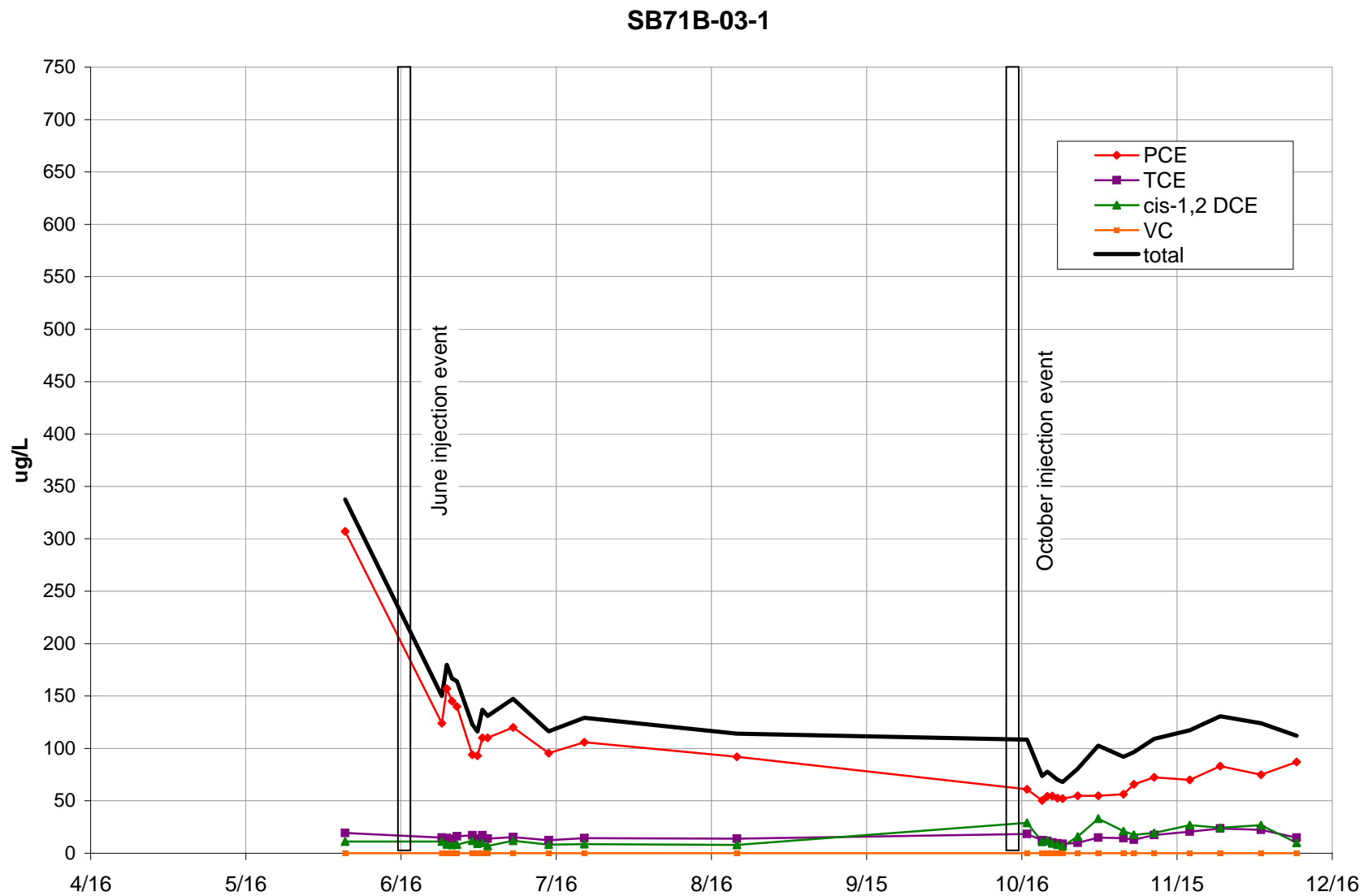


Figure 5. Changes in VOC concentrations in SB71B-03-1.

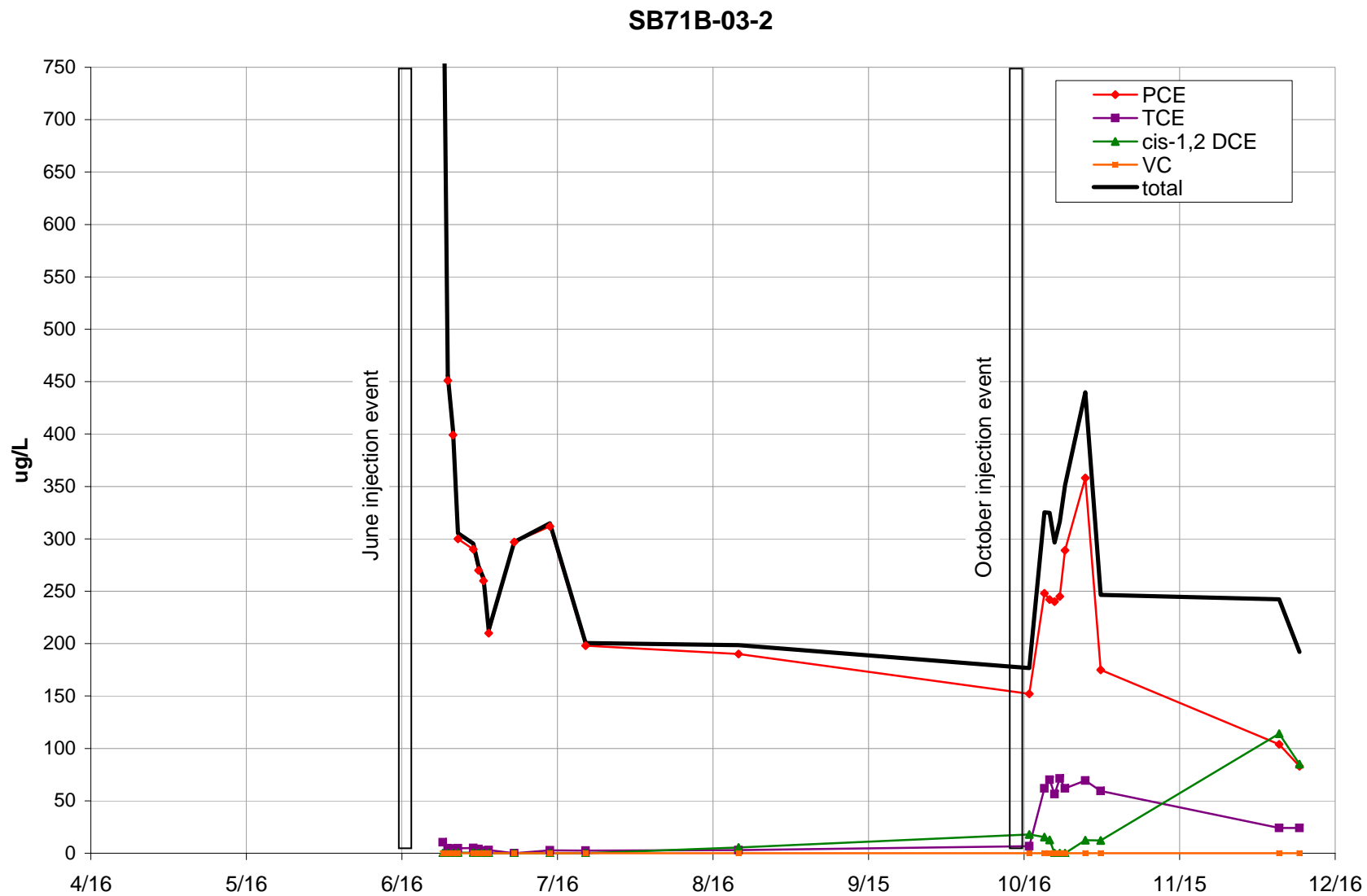


Figure 6. Changes in VOC concentrations in SB71B-03-2.

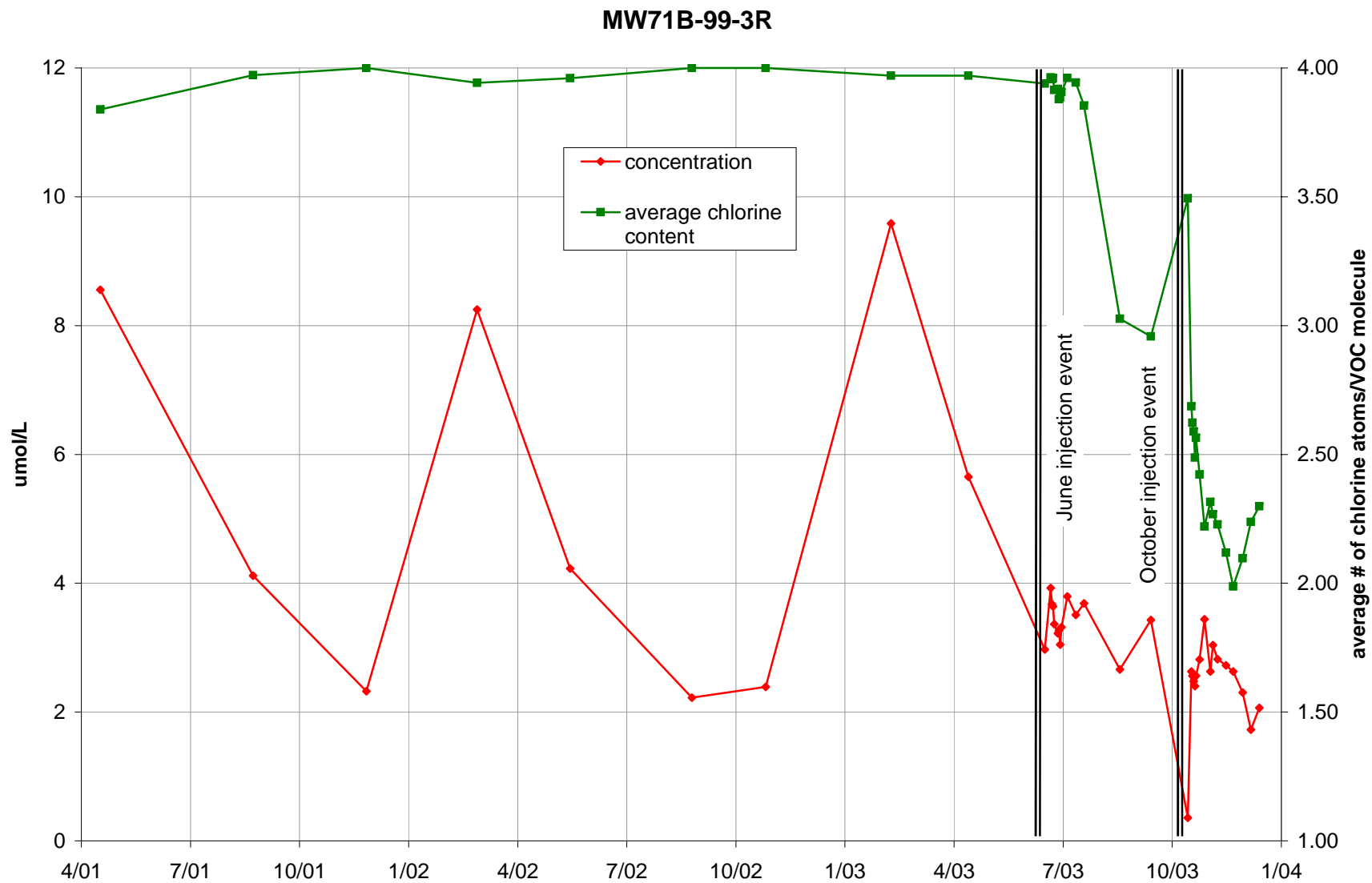


Figure 7. Long-term, molar VOC concentration changes in MW71B-99-3R.



E.O. Lawrence Berkeley National Laboratory
University of California
Environmental Restoration Program



United States Department of Energy

BUILDING 51L *IN-SITU* CHEMICAL OXIDATION PILOT TEST

for the
Lawrence Berkeley National Laboratory
Environmental Restoration Program

August 2004

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*A Joint Effort of
Environment, Health and Safety Division and
Earth Sciences Division
Lawrence Berkeley National Laboratory
Berkeley, CA 94720*

August 2004

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This work was done at the Lawrence Berkeley National Laboratory, which is operated by the University of California for the U. S. Department of Energy under contract DE-AC03-76SF00098.

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SECTION 1

SUMMARY

During the weeks of September 22, 2003 and December 8, 2003, a pilot test of *in-situ* chemical oxidation (ISCO) of chlorinated volatile aliphatic hydrocarbon contaminants (VOCs) in the engineered fill in the vicinity of Building 51L was performed via injection of citric acid and hydrogen peroxide according to the “Workplan for *In-Situ* Chemical Oxidation Pilot Test” dated May 2003 (“workplan”). The contaminants at the site consist primarily of tetrachloroethylene (PCE) and trichloroethylene (TCE) in the unsaturated zone and cis-1,2 dichloroethylene (cis-1,2 DCE) in the saturated zone.

The reagents were injected into a deep interval in two wells during the September event, and into shallow and deep intervals in 12 wells in the December injection event. The 12 injection wells in the second event were arranged in a hexagonal array with a 5-foot spacing. This array enclosed three groundwater wells.

The injection was performed by Rejuvenate under the supervision of personnel from Lawrence Berkeley National Laboratory’s (LBNL’s) Environmental Restoration Program (ERP) and Parsons Engineering. Concentrations and volumes of reagents injected into each group of wells were recorded along with the injection pressures at each well. The effect of the injection was monitored in real time via measurements of pH in the groundwater wells and observations of the seepage patterns.

Injection pressures were successfully limited to avoid hydraulic fracturing. Reagents did not seep into an adjacent storm drain or migrate into a shallow, laterally extensive sand layer at the site. Seepage during the test was primarily from open wells and the drain in the well box of monitoring well MW51L-01-4. All of this seepage was contained on the ground surface and transferred to 55-gallon drums by vacuum. No seepage migrated overland to outside the pilot test area, and no seepage entered the storm drain catch basin in the vicinity of the pilot test.

The pattern of seepage and the pH measurements indicate that a radius of influence of at least 3 feet was achieved around each well. However, this only required injection of 13% of the total reagent volume containing 15% of the acid mass and 30% of the hydrogen peroxide mass, and 26% of the total reagent volume containing 35% of the acid mass and 40% of the hydrogen peroxide mass specified in the workplan for the shallow and deep injections intervals, respectively. Based upon the response data collected, continued injection during the pilot test would likely have resulted in the additional injected volume either migrating away from the pilot test area laterally and/or seeping to the ground surface, rather than increased filling of the pore space immediately around the wells. Therefore the reagent volumes specified in the workplan were not injected due to achieving the desired radius of influence with the smaller reagent quantities, and due to the low flow rates which made injecting the workplan-specified volumes economically unfeasible.

Injection of the reagent volumes specified in the workplan would theoretically have occupied a maximum of 26% of the total pore volume around each well, which is reasonably equivalent to the likely effective porosity. The actual reagent volumes injected occupied a maximum of 3.5%, or $1/28^{\text{th}}$, and 7.5%, or $1/13^{\text{th}}$, of the total pore volume around each well in the shallow and deep intervals, respectively. The changes in pH during injection of these volumes indicate that only a small fraction of the total pore volume was accessed by the reagents. Therefore, it appears the vast majority of reagents infiltrated into and advected through significantly more permeable pathways comprising a small portion of the total soil mass. This is in accord with the distribution of soil types in the engineered fill. Well- and poorly-sorted sand make up 3% of the fill, while silty sand and gravel make up 21%. The remainder of the fill consists of silt and clay. The VOC concentration changes in the post-ISCO test soil samples support the conclusion that reagents primarily advected along coarse-grained pathways with the VOC concentration changes in the only post-ISCO soil sample containing clean, coarse-grained soil significantly more altered from the pre-ISCO concentrations than in any other sample.

Significant decreases (35% to 100%) of all volatile aliphatic hydrocarbon contaminants (VOCs) detected prior to the pilot test were measured in wells monitoring the artificial fill following the December injection event. The pattern and duration of the decreases, as well as the detection of new VOCs following the injection events and the low ratio of reagent volume to

total pore volume, strongly suggest that the decreases were due to chemical oxidation rather than dilution or some other process. As soil contamination occurs in both the coarse- and fine-grained soils, the post-test rebound is likely due to advection and diffusion of contaminants from the fine-grained soils. Therefore injection methods which could further discretize the injection interval in a full-scale ISCO relative to the pilot test could be explored. One possible method of achieving this goal is sleeve-port injection. However, injection of the necessary reagent volumes into the finer-grained soils through greater discretization is likely to be economically unfeasible due to the low injection flow rates.

SECTION 2

INTRODUCTION

During the weeks of September 22nd and December 8th, 2003, personnel from Rejuvenate conducted a pilot test of the in-situ chemical oxidation (ISCO) of chlorinated volatile aliphatic hydrocarbon contaminants (VOCs) in the subsurface adjacent to and beneath the western side of Building 51L. The objectives of this test, as stated in the workplan, were to determine if ISCO could effectively reduce the contaminant mass in soil and groundwater in the vicinity of Building 51L. This work was performed under the direction of personnel from LBNL's ERP and Parsons Engineering.

Note that the pilot test described in the workplan did not specify injection of iron along with the other reagents. Analysis of the iron content of a drill-cutting sample of the artificial fill in the vicinity of Building 51L and of several liner samples of artificial fill and colluvium in the vicinity of Building 71B indicated that the artificial fill at Building 51L had sufficient iron to allow for ISCO without the addition of iron.

SECTION 3

INJECTION WELLS

The September and December injection events were conducted in the same hexagonal, three-row array of 12 injection wells on a 5-foot spacing that enclosed a soil volume containing three temporary groundwater monitoring wells as shown on Figure 1. The eastern row consisted of four injection wells plunging 60 degrees from horizontal beneath the western edge of Building 51L. These wells are numbered IW51L-03-1 to -4 from north to south. The southern two of the declined wells bracketed temporary groundwater monitoring well SB51L-02-3. The remaining injection wells were vertical. Temporary groundwater monitoring well SB51L-03-1 and -2 were approximately centered in the northernmost and southernmost triangles of vertical injection wells, respectively.

The injection wells consisted of ¾-inch internal-diameter, stainless-steel pipe with external threaded couplings. The screen sections consisted of the same pipe with three, approximately 3/16-inch holes drilled at equal angles around the pipe. A set of these holes was drilled at approximately 4-inch intervals along the screen. Hydraulic pressure was supplied to packers above and, where necessary, below the screen via a separate line. The outside diameter of the packers was approximately 1.7 inches. The packers consisted of a heat- and chemical-resistant rubber tube clamped to the stainless steel casing.

The injection wells were installed in 2.5-inch diameter open borings advanced by direct-push. A 2-inch internal diameter PVC casing was grouted into the upper few feet of each boring. The annulus between the PVC casing and the injection casing was sealed at the top of the PVC casing by a compression fitting in order to minimize exposure to the injectate if a packer failed in the boring during injection.

SECTION 4

SEPTEMBER INJECTION EVENT

4.1 SCHEDULE OF ACTIVITIES

On Thursday and Friday, September 25th and 26th, Rejuvenate and its direct push subcontractor, Vironex, installed injection wells IW51L-03-1 to -12 at Building 51L. Injection into only wells IW51L-03-2 and -10 occurred on September 27th during this injection event.

4.2 INSTALLATION OF INJECTION WELLS

The injection wells were installed by Rejuvenate in 2.5-inch diameter borings advanced by Vironex using direct-push methods. Borings were typically advanced by hydraulic pressure on the push rod alone. Occasional hydraulic percussion was necessary to advance the rod.

4.3 INJECTION INTERVALS

During the September injection event, the packers would not hold pressure without continual flow indicating a leak somewhere in the packer system. With continual flow of 5 gpm, the pressure could be maintained at approximately 20 pounds per square inch (psi). This leak was later determined to be occurring at the well casing joints. As a consequence of this equipment failure, it was decided to maintain pressure in the packers of just two injection wells through continual flow to allow injection of some quantity of acid and hydrogen peroxide solution. This decision was taken with the realization that water leaking from the packer system would be injected somewhere into the subsurface during this process, but this was deemed acceptable in order to gather at least some data on the impact of injecting acid and hydrogen peroxide reagents into the subsurface.

Well IW51L-03-10 was chosen for injection due to its proximity to temporary monitoring well SB51L-03-1, respectively. Well IW51L-03-2 was chosen for injection as it was the closest, out of the six wells initially connected to the injection truck, to temporary monitoring well SB51L-02-3.

Well IW51L-03-2 plunged 60 degrees beneath Building 51L. The total declined depth of this well was 24 feet below ground surface (bgs; 21 feet bgs vertically). The declined depth to the artificial fill/colluvium contact was estimated as 26.5 feet bgs (23 feet bgs vertically), and the declined depth to the colluvium/Great Valley Group contact was estimated as 39 feet bgs (34 feet bgs vertically). The packers in well IW51L-03-2 were positioned for injection of the entire borehole interval below 19 feet bgs (16 feet bgs vertically).

Well IW51L-03-2 was a vertical well with a total depth of 23 feet bgs. The artificial fill/colluvium contact was estimated as 21.5 feet bgs and the colluvium/Great Valley Group contact was estimated as 28 feet bgs at the location of this well. The packers in well IW51L-03-10 were positioned for injection of the entire borehole interval below 15 feet bgs.

4.4 INJECTION PRESSURES

Prior to injection of any chemical solution, the packers were pressurized to 20 psi. Injection pressures were approximately 10 psi.

4.5 REAGENT QUANTITIES INJECTED AND REAL-TIME RESPONSES OBSERVED

Injection to well IW51L-03-10 occurred first, followed by injection to well IW51L-03-2. The depth to water (DTW) and pH prior to the commencement of injection were measured in temporary monitoring wells SB51L-02-3, SB51L-03-1 and -2, and in monitoring well MW51L-01-3. Table 1 lists the pH measurements taken during the September injection event.

Table 1. Summary of pH measurements during September injection event.

| injection to well | well | | | | | | | | | | |
|----------------------|------------|--------|-------|------------|-------|------------|--------|-------|------------|--------|-------|
| | SB51L-02-3 | | | SB51L-03-1 | | SB51L-03-2 | | | MW51L-01-3 | | |
| | before | during | after | before | after | before | during | after | before | during | after |
| IW51L-03-10 | 7.0 | | | 7.0 | 3.0 | 7.0 | | 7.0 | 7.0 | | 7.0 |
| IW51L-03-2 | | 6.5 | 6.5 | | 3.0 | | 6.5 | | | 6.5 | |

Approximately 55 gallons of reagent, consisting of 20 gallons of ~10% citric acid and 35 gallons of 17.5% hydrogen peroxide, were injected into well IW51L-03-10. The DTW was

14.35 feet below ground surface (bgs). The pH changed to 3 and the DTW rose to 1.2 feet bgs during or shortly after injection to this well. SB51L-03-1 was subsequently purged continuously throughout the injection activities with the final DTW in this well below the initial DTW.

Shortly after ceasing flow to the packers in IW51L-03-10, low pH seepage to the ground surface commenced, primarily from the drain hole inside the well box for MW51L-01-4. The timing, location and quantity of seepage indicates that the packers in the injection well were sealing the boring during the continuous flow phase, and that when flow was ceased the reagents pushed up the boring due to the off-gassing reaction, and followed a shallow lateral pathway, consisting possibly of the base rock, to MW51L-01-4 13 feet away.

Injection next took place in IW51L-03-2 with continual flow to the packers. 25 gallons of ~10% citric acid were injected. After injection of approximately 15 gallons of acid, the first pH measurement in temporary wells SB51L-02-3 and SB51L-03-2, and in monitoring well MW51L-01-3 indicated the pH had dropped 0.5 standard units. The pH in SB51L-03-2 did not change throughout the remainder of the injection. Hydrogen peroxide was not injected into this well due to concerns about the amount of water leaking from the packers.

After the injection of reagents into IW51L-03-2 and depressurization of the packers in this well, seepage was again observed from the drain hole in the MW51L-01-4 well box. 30 gallons of water were subsequently injected into IW51L-03-2 with the packers depressurized to check the connection to drain hole in MW51L-01-4 well box. Seepage from this location was found to correlate with injection. The casing in MW51L-01-4 was checked for damage and found to be intact and the DTW was measured at 22 feet bgs, indicating no intrusion of seepage into this well casing.

The effluent in the storm drain adjacent to the pilot test site was periodically monitored at a location downflow. No changes in flow rate, pH, or turbidity were observed during or after the injection event, indicating that no seepage to the storm drain occurred during injection.

4.6 DISCUSSION OF RESULTS

The workplan called for injection of 7.3 kg of citric acid in 9 gallons of solution and 37 kg of hydrogen peroxide in 97 gallons of solution per 5-foot interval for injection wells spaced 5 feet apart. 7.7 kilograms (kg) of citric acid in 25 gallons of solution and 23.7 kg of hydrogen peroxide in 35 gallons of solution were injected into the 8-foot long interval in well IW51L-03-10. Therefore, 66% of the acid mass, 40% of the hydrogen peroxide mass, and 32% of the reagent volume specified in the workplan were injected in this interval. 9.7 kg of citric acid in 25 gallons of solution were injected into the 5-foot long interval in well IW51L-03-2. This is 133% of the acid mass, 280% of the acid reagent volume, and 24% of the total reagent volume specified in the workplan. Injection was stopped short of the workplan targets due to concerns about water leaking from the packers.

The pH decrease in temporary monitoring well SB51L-03-1 during injection into well IW51L-03-10 indicates that the radius of influence in the most conductive layers during this injection was greater than 3 feet, the shortest distance from the injection well to a monitoring well.

The timing, magnitude, and position of the pH decreases during injection into IW51L-03-2 suggest that no pH response occurred during this injection. Rather the decrease of 0.5 standard pH units was due to the uncertainty of the monitoring method (pH paper). In particular, note that the pH apparently decreased in MW51L-01-3, which is screened across 15 feet of Great Valley Sequence bedrock below an aquitard consisting of colluvium. This aquitard separates VOC-contaminated groundwater in the artificial fill from groundwater with concentrations of VOCs below detection limits in the Great Valley Group below. Therefore it is unlikely that acid injected into well IW51L-03-2 reached monitoring well MW51L-01-3.

If the conclusion of no pH response during injection into well SB51L-03-2 is correct, it indicates that the radius of influence in the most conductive layers during this injection was less than 9 feet, which is the shortest distance from this injection well to a monitoring well.

The VOCs repeatedly detected in the three wells screened in the artificial fill in the pilot test area prior to injection consist of tetrachloroethylene (PCE), trichloroethylene (TCE), cis-1,2 dichloroethylene (cis-1,2 DCE), trans-1,2 dichloroethylene (trans-1,2 DCE), 1,1 dichloroethylene

(DCE), vinyl chloride (VC), and 1,1-dichloroethane (1,1 DCA) as shown in Figures 2, 3, and 4. The dominant DCE isomer in all the wells is cis-1,2 DCE. Chloroethane (CE) was consistently detected in well SB51L-03-1 after injection as shown on Figure 3. The VOCs detected in the three wells screened in the artificial fill in the pilot test area only after the injection events are chloroform, methylene chloride, methyl chloride, and methyl bromide as shown on Figures 5, 6, and 7.

One groundwater sample was collected from each of wells SB51L-02-3 and SB51L-03-1 in the week following the September injection event. Following the injection, the concentration of total DCE in SB51L-03-1, which was closest to the wells injected during this event, declined approximately 80% as shown on Figure 3. In well SB51L-02-3, the concentration of total DCE declined approximately 25% and the concentration of PCE declined 70% as shown on Figure 2. The majority of the decrease in the concentration of total DCE in this well was due to a decrease in the concentration of trans-1,2 DCE. The concentration of other VOCs, most notably 1,1 DCA in well SB51L-03-1, remained approximately constant.

The decline in concentrations of some VOCs and not others in wells SB51L-02-3 and SB51L-03-1 following the September injection event suggests the declines are due to chemical oxidation rather than dilution (which is a significant possibility owing to the unknown large quantity of water injected during this event due to leakage from the packer lines). This is additionally suggested by the presence of previously undetected VOCs in well SB51L-03-1 following the injection event, as shown on Figure 6. Three of these VOCs are only detected in the first post-injection groundwater sample collected a week from this well after the injection. This timing strongly suggests these VOCs were created by a chemical reaction resulting from the injection.

The total VOC concentration in well SB51L-03-1 increased to near background levels two months after the September injection as shown on Figure 3. The only notable change is the decrease in 1,1 DCA and the increase in CE, which is a degradation product of 1,1 DCA. This suggests the instantiation of a long-term degradation process. As shown on Figure 2, the total VOC concentration in well SB51L-02-3 decreased significantly compared to background levels after the September injection, particularly when consideration is given to the typical pre-injection pattern of increased concentrations in the dry season. Significant precipitation did not begin until mid-December in the July 1, 2003 to June 30, 2004 rain year. The total VOC concentration

decrease in SB51L-02-3 may be due to advection of treated groundwater into the vicinity of well SB51L-02-3 due to a groundwater flow towards extraction well EW51L-00-1 to the south.

During the two years prior to the September injection event, no VOCs were repeatedly detected in quarterly groundwater samples from monitoring wells MW51L-01-3 and -4, which are screened in the Great Valley Group beneath and near to the pilot test site, respectively. No VOCs were detected in the quarterly groundwater samples from monitoring wells MW51L-01-3 and -4 a month and a half after the September injection event. This indicates the September injection did not induce leakage of groundwater from the artificial fill to the Great Valley Group through the intervening colluvial aquitard.

SECTION 5

DECEMBER INJECTION EVENT

5.1 SCHEDULE OF ACTIVITIES

On Tuesday, December 9th, a direct-push rig and operator from Gregg Drilling was mobilized to the Building 51L site to assist with removing injection wells previously installed by Rejuvenate on September 25th and 26th. These wells had proven faulty during an attempt to the conduct the ISCO pilot test on September 27th. After removal of these wells by Gregg Drilling, Rejuvenate installed new wells configured for injection into the deeper interval at the site. On Wednesday, December 10th Rejuvenate connected well heads and injection lines and commenced injection. Injection in the deep interval continued on Thursday and Friday, December 11th and 12th. On the afternoon of Friday, December 12th and the morning of Saturday, December 13th, Rejuvenate pulled the injection wells, and reconfigured and reinstalled them for injection in the shallow interval. Injection of this interval was completed during the afternoon of Saturday, December 13th.

5.2 INJECTION INTERVALS

The ISCO pilot test injection was targeted to treat the engineered fill in the pilot test area at Building 51L, as described in the workplan. As previously mentioned, the injection wells were initially configured and installed for injection into the deeper interval in the engineered fill. The injection wells were subsequently reconfigured and reinstalled for injection into a shallower interval. Table 2 below lists the deeper and shallower injection intervals at each well along with the estimated depth to the engineered fill/colluvium contact and the colluvium/Great Valley Sequence contact.

Table 2. Geologic contacts and injection intervals.

| well name | depths (ft)* | | | | |
|-------------|---------------|----------------|----------------|-----------------|------------------|
| | Qf/Qu contact | Qu/Kgv contact | bottom of hole | deep interval | shallow interval |
| IW51L-03-1 | 27.5 (24) | 44 (38) | 24 (21) | 16-24 (14-21) | 6-13 (5-11) |
| IW51L-03-2 | 26.5 (23) | 39 (34) | 24 (21) | 16.5-24 (14-21) | 6-13 (5-11) |
| IW51L-03-3 | 23.5 (20.5) | 34 (29.5) | 24 (21) | 16.5-24 (14-21) | 7-14 (6-12) |
| IW51L-03-4 | 21 (18) | 28.5 (24.5) | 24 (21) | 16.5-24 (14-21) | 7-14 (6-12) |
| IW51L-03-5 | 22.5 | 27 | 23 | 16.5-23 | 6-13 |
| IW51L-03-6 | 21.5 | 26 | 23 | 16.5-23 | 6-13 |
| IW51L-03-7 | 20 | 25 | 20 | NA | 6-20 |
| IW51L-03-8 | 18 | 24 | 20 | 12.5-20 | 7-20 |
| IW51L-03-9 | 16.5 | 23 | 18 | 11-18 | NA |
| IW51L-03-10 | 21 | 28 | 23 | 16.5-23 | 6-13 |
| IW51L-03-11 | 19.5 | 27 | 20 | 13-20 | 5-12 |
| IW51L-03-12 | 18 | 25 | 20 | 13-20 | 5-12 |

* depths in parentheses are vertical equivalents of declined depths

The bottom depth of each boring was selected so as to penetrate the colluvium beneath the engineered fill in order to assure the entire base of the fill section was treated. The base of each injection boring was also selected so as to separate it from the top of the relatively permeable Great Valley Sequence beneath by at least three feet of relatively lower permeability colluvium. This was done in an effort to prevent the injected solutions from preferentially flowing into the Great Valley Sequence.

The workplan for the ISCO pilot test specified 5-foot long injection intervals. Despite repeated communication with Rejuvenate regarding this requirement, the contractor arrived on site with 6-foot long injection screens. When combined with blank casing stubs on either side of the screen associated with either a packer unit or an end cap, the minimum length of an injection interval was 6.5 feet. Due to the necessity to complete the pilot test at the appointed time, the decision was made in the field to accept this length of injection interval.

Due to concerns about the inability of the packers to seal against a previously injected borehole wall, the depths of the shallow injection intervals were selected such that the top of the packer below the screen would inflate against an uninjected section of borehole wall. This decision resulted in an uninjected interval ranging from 1 to 3.5 feet in length between the deep and shallow intervals. Further, this decision, combined with the greater than expected length of the injection

intervals, led to selection of two 7-foot long intervals rather than the three 5-foot intervals originally envisioned based upon the workplan.

Packers could not be placed below a depth of approximately 12 feet in IW51L-03-7 due to the presence of a piece of packer torn off from the previously installed well. Therefore both the shallow and deep intervals were injected simultaneously during injection of the shallow intervals in the other wells. This was achieved by placing a packer above the shallow interval in IW51L-03-7 with no packer below.

A clean sand layer exists between 2 and 5 feet deep at injection wells IW51L-03-4 and -9. Based upon previous borings, the lateral margins of this sand layer lie approximately 10 feet to the east, 20 feet to the west and 80 feet to the south. This layer potentially has significantly higher permeability than the underlying, predominantly fine-grained soils. As such, this layer had the capacity to absorb a large amount of reagent and transmit these reagents a significant distance from the pilot test site. In addition to absorbing and transmitting reagents away from the target volume, reagents migrating through this layer could potentially contact a cast-iron storm drain passing beneath Building 51L causing increased corrosion of this pipe. Due to these concerns, the following measures were taken to forestall the entry of reagents to this layer.

The top of the shallow injection interval in IW51L-03-8 was set to a depth of 7 feet to assure at least two feet of relatively less permeable fine-grained engineered fill between the top of the injection interval and the base of the nearby sand layer. It was not possible to inflate a packer against uninjected borehole wall below the shallow interval in this well as the top of the deeper interval was 12.5 feet. Therefore, as with IW51L-03-7, only a single packer was placed above the shallow interval and the shallow and deep intervals were injected simultaneously during injection of the shallow intervals in the other wells. The shallow interval in IW51L-03-9 was not injected due to the small additional borehole length which could potentially be injected above the deep interval (7 to 11 feet deep), and the risk that reagents injected in this interval might migrate into the clean sand layer. Finally, a 6-foot deep boring was installed through the sand layer 4 feet south of IW51L-03-9. This boring was left open during the injection activities to monitor for migration of reagents into and through the sand layer.

5.3 INJECTION PRESSURES

Based upon the instability of the hydrogen peroxide, chemical oxidation using this reagent is generally believed to be effective for only a matter of hours after injection. Therefore, advection driven by injection pressure must bring the reagents into contact with the target contaminants to be oxidized within a very short time period. This is unlike more persistent in-situ treatment technologies where natural groundwater advection may be relied upon to bring the reagents, or their byproducts, into contact with the target molecules. The above suggests that injection pressures must be controlled carefully to prevent hydraulic fracturing of the formation that would cause the reagents to bypass most of the pore volume containing the target contaminants.

Hydraulic fracturing from vertical wells in the shallow subsurface is generally related the vertical stress in the material around the well, which is typically equal to the overburden pressure. The minimum overburden pressure in each injection interval occurs at the shallowest portion of the interval. This pressure was calculated based upon a review of dry densities and moisture contents from geotechnical reports and water level measurements from ERP wells in the engineered fill near Building 51L.

The average dry density of the engineered fill was 103 pounds per cubic foot. The average moisture content was 17% above a depth of approximately 8 feet, which equates to a saturation of 80% (assuming a specific density for the solids of 2.65). Below approximately 8 feet the saturation is 100%. The average water level in the area is 13 to 14 feet deep, indicating a five to six foot capillary fringe. Using the numbers above, the minimum overburden pressure was calculated as 10 to 11 pounds per square inch (psi) in the deep interval and 5 to 6 psi in the shallow interval. The maximum injection pressures were set at 2 to 2.5 times the minimum overburden pressures in order to maximize injection pressure-driven advection within safe limits. This multiplier is based upon the general field experience pressures to initiate hydraulic fractures are significantly greater than overburden pressures, in part due to soil cohesion. Therefore the maximum target injection pressure for the deep interval was 24 psi and for the shallow interval was 12 psi.

Significant expansion of the packers occurs at approximately 15 psi. This conclusion was based upon discussions with Rejuvenate as well as direct observation of partial inflations of packers laid out on the ground surface. This suggests only the increment of packer pressure above 15 psi is effective at resisting passage of injected reagents. During injection, the target packer pressure was set at 15 psi higher than the maximum target injection pressures, or approximately 40 psi for the deep interval and 25 psi for the shallow interval. Other than the one instance described below, the injection pressures and packer pressures were maintained according to the specifications described above.

The effectiveness of the 15 psi pressure differential between the packer and injection pressures was confirmed during the first round of deep interval injection. During this injection there was no seepage at the ground surface when the pressure differential was greater than 15 psi. At the end of the injection the differential decreased to less than 5 psi due to a decision by Rejuvenate to increase the injection pressure without increasing the packer pressure. Rejuvenate took this action without prior discussion with ERP personnel in an attempt to increase the injection rate. Shortly thereafter seepage commenced from the nearby drain inside the MW51L-01-4 well box, indicating migration of reagents up to and through the shallow subsurface. ERP personnel recommended a reduction in injection pressure as soon as the pressure increase was observed. However, the injection was complete at this time. Approximately 5 gallons of reagent were injected at the higher pressure.

5.4 INJECTION INTERVAL PERMEABILITY

Rejuvenate's injection truck includes two approximately 100 gallon tanks for acid and water and an approximately 200 gallon tank for hydrogen peroxide solution. These tanks are connected via valved piping to a single pump. Fluid exiting the pump can be directed to circulate back to the originating tank or to a manifold with three valved ports which afford independent pressure control to three external hoses. Each external hose can connect to one or two injection wells. The injection truck also includes a separate pump for inflating and maintaining pressure in the packers.

The pump truck includes various types of flow meters between the injection pump and manifold and within the manifold. None of these flow meters could be shown to be quantitatively accurate to the satisfaction of the ERP personnel supervising the pilot test. Therefore direct readings of reagent tank fluid levels combined with elapsed times were employed to gauge injection rates.

The lack of quantitatively reliable flow meters on each injection line made it necessary to initially inject each well interval singly in order to qualitatively gauge permeability. Each interval in each well was injected at equal to or less than the maximum injection pressure for fifteen minutes or until approximately 4 gallons of acid had been injected. The first 2 gallons injected were sufficient to fill the 6 to 7 foot-long boring interval with reagent. Fifteen minutes to inject an additional 2 gallons into the formation (equivalent to 0.15 gpm) was selected as it is a lower bound for the economic feasibility of *in-situ* treatment via reagent injection. At this rate, approximately 70% of the reagent volume specified in the workplan could be injected in an 8-hour period.

Injection pressures at the truck manifold and the well head provided secondary confirmation of the permeability around well. A well head pressure less than or equal to the manifold pressure on the same injection line indicated flow to the well (the manifold pressure gauges were typically positioned approximately 4 feet above the well head pressure gauges). A well head pressure less than the maximum injection pressure also typically indicated flow to the well. Relative differences in the flow rate to each well during multiwell injection, as measured qualitatively by flow meters on the injection manifold, provided a tertiary confirmation of differences in the permeability around each well.

Based upon data accumulated via the three methods outlined above, the deep injection intervals in IW51L-03-2, -4, -5, -8, -10, and -12, or approximately half all the wells, were sufficiently permeable to accept 2 gallons of reagent within 15 minutes. Intervals which passed this test were not distributed according to any discernible pattern; rather they were randomly distributed among the intervals which failed this test. All of the shallow injection intervals were sufficiently permeable to accept 2 gallons of reagent within 15 minutes. The permeability difference between shallow and deep intervals could perhaps be due to differences in

consolidation of the fill and/or differences in saturation. Geologic logs in the area do not indicate significant differences in grain-size in the shallow injection interval versus the deep injection interval.

5.5 REAGENT QUANTITIES INJECTED AND REAL-TIME RESPONSES OBSERVED

Approximately 430 gallons of reagent were injected in the deep interval. This consisted of 95 gallons of 10% citric acid, 75 gallons of 12.5% hydrogen peroxide solution, and 260 gallons of 17.5% hydrogen peroxide solution. These reagents were injected in approximately four equal volumes during four periods over the course of three days, as listed in Table 3. The total elapsed injection time was 5 hours, and therefore the average injection rate to six wells at a time was 1.5 gallons per minute (gpm), or 0.25 gpm per well.

Table 3. Wells injected during each injection period

| Injection | | | IW51L-03 wells injected during period |
|-----------|-----------------|--------------------------|---------------------------------------|
| No. | period | interval | |
| 1 | 12/10 afternoon | northern wells - deep | 1, 2, 5, 6, 10 |
| 2 | 12/11 morning | northern wells - deep | 1, 2, 3, 5, 6, 10 |
| 3 | 12/11 afternoon | southern wells - deep | 11, 3, 4, 8, 9, 12 |
| 4 | 12/12 morning | southern wells - deep | 11, 3, 4, 8, 9, 12 |
| 5 | 12/13 afternoon | southern wells - shallow | 7, 8, 3, 4, 11, 12 |
| 6 | 12/13 evening | northern wells - shallow | 1, 2, 5, 6, 10, 11 |

Approximately 210 gallons of reagent were injected in the shallow interval. This consisted of 30 gallons of 10% citric acid and 180 gallons of 17.5% hydrogen peroxide solution. These reagents were injected in approximately two equal volumes during two periods in one day, as listed in Table 2. The total elapsed injection time was 1 ½ hours, and therefore the average injection rate to six wells at a time was 2.3 gallons per minute (gpm), or 0.4 gpm per well.

Packer failures occurred four times during injection. These failures were observed as a precipitous drop in the well head injection pressure. Both the packer and reagent feed lines to the well were shut off at the wellhead in these instances. A packer failed in IW51L-03-5 near the

midpoint of the second injection period and in IW51L-03-10 at the end of the second injection period. A packer in an undiagnosed position failed at the end of the fourth injection period. A packer in IW51L-03-2 failed near the beginning of the sixth injection period. The only failed packer observed upon well removal was in IW51L-03-10. The upper packer failed by splitting longitudinal along most of its length. The shape of a notch/dimple along the split edge suggested that the packer had impinged on something sharp in the borehole, such as a gravel grain.

The pH in three temporary monitoring wells area screened in the engineered fill in the pilot test area was measured prior to commencement of injection and periodically throughout the injection. These measurements are summarized in Table 4. The pH of the effluent in a storm drain passing near the pilot test area was also measured to monitor for potential seepage of injected reagents into the storm drain. This storm drain consists of a 24-inch diameter, reinforced concrete pipe whose invert is located approximately 4 feet west of the pilot test area and at a depth of approximately 15 feet.

Table 4. Summary of pH measurements during injection.

| Injection | | | Well | | | | | | Storm Drain below pilot test site |
|-----------|-----------------|--------------------------|------------|-------|------------|-------|------------|-------|---|
| | | | SB51L-02-3 | | SB51L-03-1 | | SB51L-03-2 | | |
| No. | period | interval | before | after | before | after | before | after | |
| 1 | 12/10 afternoon | northern wells - deep | 6.5 | 6.5 | 6.5 | 6 | 6.5 | | 6.5 |
| 2 | 12/11 morning | northern wells - deep | 6.5 | 6.5 | 5 | 3.5 | 6.5 | | 7 |
| 3 | 12/11 afternoon | southern wells - deep | 2 | | | | 3.5 | | 7 |
| 4 | 12/12 morning | southern wells - deep | 3 | | 3 | | 2 | | 7 |
| 5 | 12/13 afternoon | southern wells - shallow | 3.5 | 5 | 3 | 3 | 4 | 3 | 7 |
| 6 | 12/13 evening | northern wells - shallow | 3 | | 3 | | 3 | | |

The pH of the storm drain effluent was measured at the closest access to the storm drain downflow of the pilot test area. This location is approximately 230 feet northwest of the pilot test area where the storm drain emerges on the slope and transitions to an 18-inch diameter corrugated metal pipe. Access to the effluent consists of a hinged hatch in the top of the pipe. The pH of the effluent indicated no seepage occurred into the storm drain during the pilot test.

Seepage of reagents was observed at a variety of locations during injection. This seepage was typically contained on the surface with sand-filled rubber socks, if necessary, and then vacuumed into 55-gallon drums. The timing and location of the seepage is listed below in Table 5, as well as any actions taken to minimize the seepage. In addition to those actions listed, the packers were pressurized in IW51L-03-2 during the fourth injection period, in IW51L-03-9 during the fifth injection period, and in IW51L-03-3 during the sixth injection period in order to minimize potential seepage from these wells.

Table 5. Summary of seepage timing and location.

| injection | | | seepage | | | |
|-----------|-----------------|--------------------------|---------------------------------|------|------------|--------------------------|
| No. | period | interval | location | time | amount | action |
| 1 | 12/10 afternoon | northern wells - deep | drain in MW51L-01-4 well box | end | 5 gallons | lower injection pressure |
| 2 | 12/11 morning | northern wells - deep | pavement joints near MW51L-01-4 | end | gas | |
| 3 | 12/11 afternoon | southern wells - deep | IW51L-03-2 | mid | 25 gallons | shut off IW3, 11, 12 |
| 4 | 12/12 morning | southern wells - deep | IW51L-03-1 | mid | 15 gallons | reduced IW3, 11, 12 |
| | | | SB51L-03-2 | mid | foam | |
| 5 | 12/13 afternoon | southern wells - shallow | IW51L-03-1 | mid | minimal | shut off IW3, 7, 11 |
| | | | IW51L-03-2 | mid | continuous | shut off IW3, 7, 11 |
| 6 | 12/13 evening | northern wells - shallow | drain in MW51L-01-4 well box | mid | minimal | |
| | | | SB51L-03-1 | end | minimal | |

No seepage was observed into the 6-foot deep boring through the sand layer south of IW51L-03-9 indicating reagents did not enter this layer in significant quantities.

5.6 DISCUSSION OF RESULTS

The seepage and injection pressure data do not indicate that hydraulic fracturing occurred, with one possible exception. The seepage pattern from IW51L-03-1 and -2 during deep interval injection suggests hydraulic fracturing may have occurred in this area. The pressure data from these wells and the surrounding wells IW51L-03-3, -5 and -6, however, do not record any decrease in the injection pressures during the first and second injection periods.

Such a pressure drop would be expected if one or more hydraulic fractures were initiated from these wells. Therefore it appears unlikely that hydraulic fracturing occurred, and the pattern of seepage was likely due to some pre-existing “fast path.”

The workplan called for injection of 7.3 kg of citric acid in 9 gallons of solution and 37 kg of hydrogen peroxide in 97 gallons of solution per 5-foot interval. The average quantities injected in each, approximately 7-foot long, deep interval were 3.5 kg of citric acid in 8.5 gallons of solution and 19.5 kg of hydrogen peroxide in 30.5 gallons of solution. Therefore 35% of the acid mass, 40% of the hydrogen peroxide mass, and 26% of the total reagent volume specified in the workplan was injected in the deep interval. The average quantities injected in each, approximately 7-foot long, shallow interval were 1.0 kg of citric acid in 2.5 gallons of solution and 11 kg of hydrogen peroxide in 16.5 gallons of solution. Therefore 15% of the acid mass, 30% of the hydrogen peroxide mass, and 13% of the reagent volume specified in the workplan was injected in the shallow interval.

The masses and volumes specified in the workplan were not achieved due to the low flow rates at the maximum allowable injection pressures. At these flow rates, injection of the workplan specified volumes would have taken 109 hours and 68 hours of elapsed injection time in the deep and shallow intervals. This length of time was not economically feasible for the pilot test, and indicates that full-scale treatment would not be economical either.

Based upon dry density and moisture content measurements in geotechnical reports and water level measurements, the engineered fill in the vicinity of Building 51L has an average dry density is 102.5 pounds/cubic foot (lbs/ft³) and an average total porosity of 38%. Therefore the pore space within a 3-foot diameter cylinder around each 7-foot long injection interval is 75 cubic feet. However, the pH measurements and seepage patterns indicate that only 5.5 and 2.5 cubic feet of reagent injected per well in the deep and shallow injection intervals, respectively, were required to reach a radius of influence of 3 feet or more. As these reagent solution volumes are only 7.5% and 3.5% of the total pore volumes respectively, this suggests the radius of influence was due to flow through pathways which were significantly more permeable than the average, and which occupied only a small percent of the total soil volume.

Detailed logging of the engineered fill in the vicinity of Building 51L was only performed on the one core from SB51L-01-9. This log indicates that the engineered fill consists of 3% well and poorly sorted sand (SW and SP) and 21% silty sand and gravel (SM and GM) by volume. The remaining volume consists of clay and silt (predominantly CL and ML). Based upon the inferred radius of influence, the total volumes injected, and the proportion of the engineered fill consisting of coarse-grained soil, it is reasonable to presume that the majority of the reagents infiltrated and advected through the coarse-grained soils with only minimal infiltration into the fine-grained soils.

Comparison of the reagent to soil mass ratios specified in the workplan (1:1000 for citric acid, 1:200 for hydrogen peroxide) with the ratio of injected reagent mass to coarse-grained soil mass within 3 feet of each injection interval (1:4300 for citric acid and 1:390 for hydrogen peroxide in the shallow interval, 1:1220 for citric acid and 1:230 for hydrogen peroxide in the deep interval) suggests contaminants residing in the coarse-grained soils were not completely oxidized in the shallow interval and were not completely oxidized in the deep interval during the pilot test. The unsaturated zone approximately coincides with shallow interval and the deep interval approximately coincides with the saturated zone. Therefore, significant, short-term declines in groundwater contaminant concentrations should be observed as the coarse-grained soils are more permeable and provide the majority of the water in any well sample. Significant rebound of groundwater contaminant concentrations should follow due to a lack of oxidation of contaminants in the less permeable, fine-grained soils within which soil sample results indicate contaminants are also present.

Following the December injection event, the VOC concentrations in all three wells screened in the artificial fill within the pilot test area decreased significantly as shown on Figures 2, 3, and 4. The total VOC concentration decreased approximately 80%, 35% and 100% in wells SB51L-02-3, SB51L-03-1 and SB51L-03-2, respectively. The concentrations remain significantly decreased for almost two weeks following injection in wells SB51L-03-1 and -2, and for almost four weeks in well SB51L-02-3. Following these periods, concentrations rapidly return to pre-injection levels.

Three previously undetected VOCs are measured in the first post-injection groundwater sample (collected three days after injection) from wells SB51L-02-3 and SB51L-03-2, as shown on Figure 5 and 7. This timing strongly suggests these VOCs were created by a chemical reaction resulting from the injection. Additionally, these VOCs are the same as those detected in SB51L-03-1 after the September injection.

The VOC concentration decreases in all three wells following the December injection indicate that either dilution or oxidation of these contaminants occurred. The persistence of the concentration decreases and the generation of new VOC constituents indicate that oxidation was probably the dominant process. The magnitude of the decreases relative to the low ratio of reagent volume to total pore volume further suggests that oxidation rather than dilution was the dominant process. The rapid rebound of concentrations following the December injection is probably due in part to the beginning of significant precipitation in mid-December. Water level measurements in 2001 from an extensive temporary well array in the area indicated precipitation infiltrated through pavement cracks in the vicinity of Building 51L and recharged the saturated zone.

The concentration decrease in SB51L-03-1 is less than in the other two wells following the December event, and there were no new VOC constituents in this well after the December event. The concentration decline and new VOC generation in SB51L-03-1 after the September injection were as significant as those in the other two wells after the December injection, suggesting the chemistry around this well was altered by the September injection event. The availability of endogenous iron may have been significantly reduced following the September event by citrate chelation and subsequent advection away from the well. Reduction in the available iron would have reduced the effectiveness of the December injection, particularly as no iron was injected. Calculation of the average linear velocity could provide some perspective on the likelihood of these hypotheses, however this velocity cannot be estimated from the available water level data. The hydrographs from wells SB51L-02-3 and SB51L-03-1 and -2 are too irregular to confidently ascertain the water table position within a few months of the injection events. Additionally, the coverage provided by these wells may not be sufficient to determine the gradient within the pilot test area accurately, as demonstrated by the spatial variability of water levels in a denser array of temporary wells in 2001.

No VOCs were detected in the quarterly groundwater samples from monitoring wells MW51L-01-3 and -4 a month and a half after the December injection event. This indicates the December injection did not induce leakage of groundwater from the artificial fill to the Great Valley Group through the intervening colluvial aquitard.

SECTION 6

SOIL RESULTS

Soil samples were collected from soil borings SB51L-04-1 and -2 within the ISCO test area on March 5th, 2004 as shown on Figure 8. Soil samples of the artificial fill from borings SB51L-04-1 and -2 were collected from within half a foot of the depth of pre-ISCO test soil samples from adjacent borings SB51L-01-9 and -3, respectively, as shown on Figure 9. All of these samples were collected by direct-push coring using plastic liners. Comparisons of the most commonly detected VOCs in the soil samples (PCE, TCE, and cis-1,2 DCE) are shown on Table 6 and Figure 10.

The total mass of PCE and TCE after the ISCO test compared to prior to the test was approximately the same while the total mass of cis-1,2 DCE doubled as shown by the sums in Table 6. The ratio of post- to pre-ISCO concentrations for each compound for each sample pair is shown on Figure 11 (only sample pairs with no non-detects are shown). For all sample pairs, the TCE concentration ratio is either within or near the range of the PCE and cis-1,2 DCE ratios.

All of the sample pairs taken at exactly the same depth have a cis-1,2 DCE concentration ratio higher than the PCE and TCE ratios, which is consistent with reductive dechlorination of either in-situ, or mobilized, PCE and TCE. Sample pairs with mismatched depths have the reverse: a cis-1,2 DCE concentration ratio lower than the PCE and TCE ratios. The coincidental probability of this correlation is 1:84, therefore it is likely the cause of the reverse ratios is the sample depth difference. The pre-test concentrations may not have been equivalent at the different depths and/or there might have been differing responses to the ISCO test at different depths.

Sums of the same-depth sample pair results indicate significant reduction in PCE, some reduction in TCE, and more than a doubling in cis-1,2 DCE by mass. Due to the elapsed time between the sample dates, it is difficult to discern if the concentration differences observed are due to naturally occurring reductive dechlorination or due to reductive dechlorination initiated by injection of the chemical oxidation reagents, as was observed at the Building 71B ISCO test. However, the observed concentration changes appear too large to occur naturally during the

approximately 3 years between sample collection, as back extrapolation from this rate would imply there was free-phase solvent at the site decades ago, which is not in accord with the groundwater plume shape or concentrations. Therefore, it is more likely that the changes are due to reductive dechlorination initiated by injection of the chemical oxidation reagents, as was observed after the ISCO test at Building 71B. One hypothesis is that the injected citrate was utilized as a growth substrate by the endogenous microbial community.

The sample pair at 5 ft bgs from SB51L-01-9 and -04-1 has the second largest absolute decrease in PCE, the largest absolute decrease in TCE, the largest absolute increase in cis-1,2 DCE, and the smallest post- to pre-test PCE and TCE ratios as shown on Figure 11. This sample pair accounts for most of the TCE mass reduction and most of the cis-1,2 DCE mass increase in the same depth sample pair set. The post- to pre-test cis-1,2 DCE ratio to PCE ratio is also larger than for any other sample pair by almost an order of magnitude, as suggested by the steeper slope for this sample pair on Figure 11. Therefore this sample pair appears to have experienced the most reductive dechlorination of any of the sample pairs. This sample pair was also the only pair to include clean, coarse-grained soil. As previously mentioned, the results of the Building 71B ISCO test indicated that ISCO could induce reductive dechlorination. The results from this sample pair therefore tend to confirm the conjecture that the injected reagents preferentially advected through the coarse-grained soils.

Table 6. Soil sample results from prior and after the ISCO test.

| | | concentration (mg/kg) | | | | | | | | | | | |
|---------------------------------------|-------------|-----------------------|--------|--------|--------|-------|------|-------|--------|-------------|-------|--------|--------|
| | | PCE | | | | TCE | | | | Cis-1,2-DCE | | | |
| | | pre | post | delta | %delta | pre | post | delta | %delta | pre | post | delta | %delta |
| SB51L-01-9 & -04-1 | 2 | 0.19 | 0.0086 | -0.181 | -95% | 0.59 | 0.14 | -0.45 | -76% | 0.041 | 0.027 | -0.014 | -34% |
| SB51L-01-9 & -04-1 | 5 | 0.25 | 0.0083 | -0.242 | -97% | 3.6 | 0.79 | -2.81 | -78% | 0.55 | 1.9 | 1.35 | 245% |
| SB51L-01-9 & -04-1 | 8.5 & 8.9 | 0.29 | 0.37 | 0.08 | 28% | 0.73 | 0.67 | -0.06 | -8% | 0.18 | 0.1 | -0.08 | -44% |
| SB51L-01-9 & -04-1 | 12.5 & 12.2 | 0.18 | 1.1 | 0.92 | 511% | 0.51 | 1.8 | 1.3 | 253% | 0.028 | 0.13 | 0.1 | 364% |
| SB51L-01-9 & -04-1 | 16.5 | 0.009 | 0.06 | 0.051 | 567% | 0.24 | 1.3 | 1.1 | 442% | 0.012 | 0.34 | 0.33 | 2733% |
| SB51L-01-3 & -04-2 | 2.3 | <0.005 | <0.005 | 0 | 0% | 0.023 | 0.1 | 0.1 | 335% | 0.032 | 0.019 | -0.013 | -41% |
| SB51L-01-3 & -04-2 | 5 | <0.005 | <0.005 | 0 | 0% | 0.55 | 0.12 | -0.43 | -78% | 0.036 | 0.012 | -0.024 | -67% |
| SB51L-01-3 & -04-2 | 8.5 & 8.8 | 0.015 | 0.025 | 0.01 | 67% | 0.79 | 1.3 | 0.5 | 65% | 0.11 | 0.051 | -0.059 | -54% |
| SB51L-01-3 & -04-2 | 12.5 | 0.34 | 0.048 | -0.292 | -86% | 0.34 | 0.65 | 0.3 | 91% | 0.066 | 0.08 | 0.01 | 21% |
| SB51L-01-3 & -04-2 | 16.5 | 0.005 | <0.005 | 0 | 0% | 0.49 | 1.3 | 0.8 | 165% | 0.04 | 0.14 | 0.1 | 250% |
| SB51L-01-3 & -04-2 | 20.5 | 0.021 | <0.005 | -0.016 | -76% | 0.83 | 0.25 | -0.58 | -70% | 0.37 | 0.16 | -0.21 | -57% |
| Sum | | 1.31 | 1.6399 | 0.33 | 25% | 8.693 | 8.42 | -0.27 | -3% | 1.465 | 2.959 | 1.49 | 102% |
| Sum of same depth pair results | | 0.825 | 0.145 | -0.680 | -82% | 6.66 | 4.65 | -2.01 | -30% | 1.15 | 2.68 | 1.53 | 133% |

SECTION 7

CONCLUSIONS

The workplan was followed to the extent practicable and economical given the low permeabilities encountered at the Building 51L pilot test site. Based upon changes in the concentrations of VOCs in the wells monitoring the artificial fill within the pilot test area, and the low ratio of reagent volume to total pore volume, ISCO significantly reduced VOC concentrations through oxidation rather than dilution.

The volumes injected represented a small fraction of the total pore space within the pilot test area. These volumes are consistent with injection primarily into the coarser-grained soils in the pilot test volume. Observations of the pH changes during injection are consistent with the hypothesis that reagents primarily infiltrated these “fast paths.” The results of pre- and post-ISCO soil sampling are also generally consistent with this hypothesis. As significant VOC contaminant mass resides within both the coarse- and fine-grained soils, rebound of VOC concentrations after a full-scale ISCO due to advection and diffusion of untreated contaminants residing in fine-grained soils is probably a significant limitation on the success of this remedial method. Discretizing the contaminated zone into a greater number of injection intervals during full-scale ISCO as compared to the two intervals used during the pilot test might increase the effectiveness of ISCO. However, ISCO would probably still fail to reduce contaminant concentrations in the long term even with increased discretization due to the even lower permeabilities, and therefore flow rates, that would be encountered in fine-grained-only injection intervals.

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- Figure 11. Comparison of PCE, TCE, and Cis 1,2-DCE concentration ratios for each pre- and post-ISCO test soil sample pair. Sample sets with nondetects are not shown.

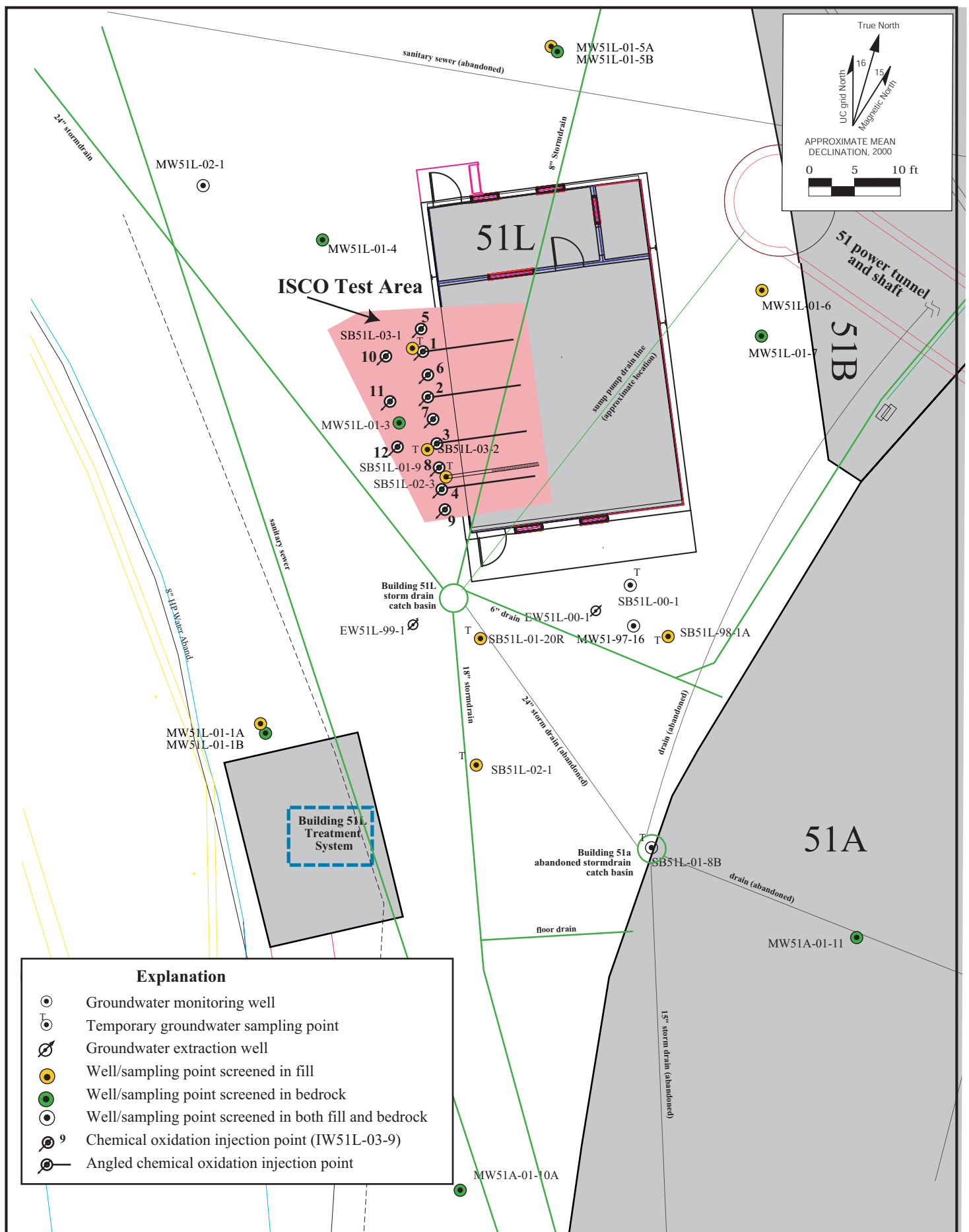


Figure 1. Locations of in-situ chemical oxidation (ISCO) pilot test area at Building 51L.

SB51L-02-3

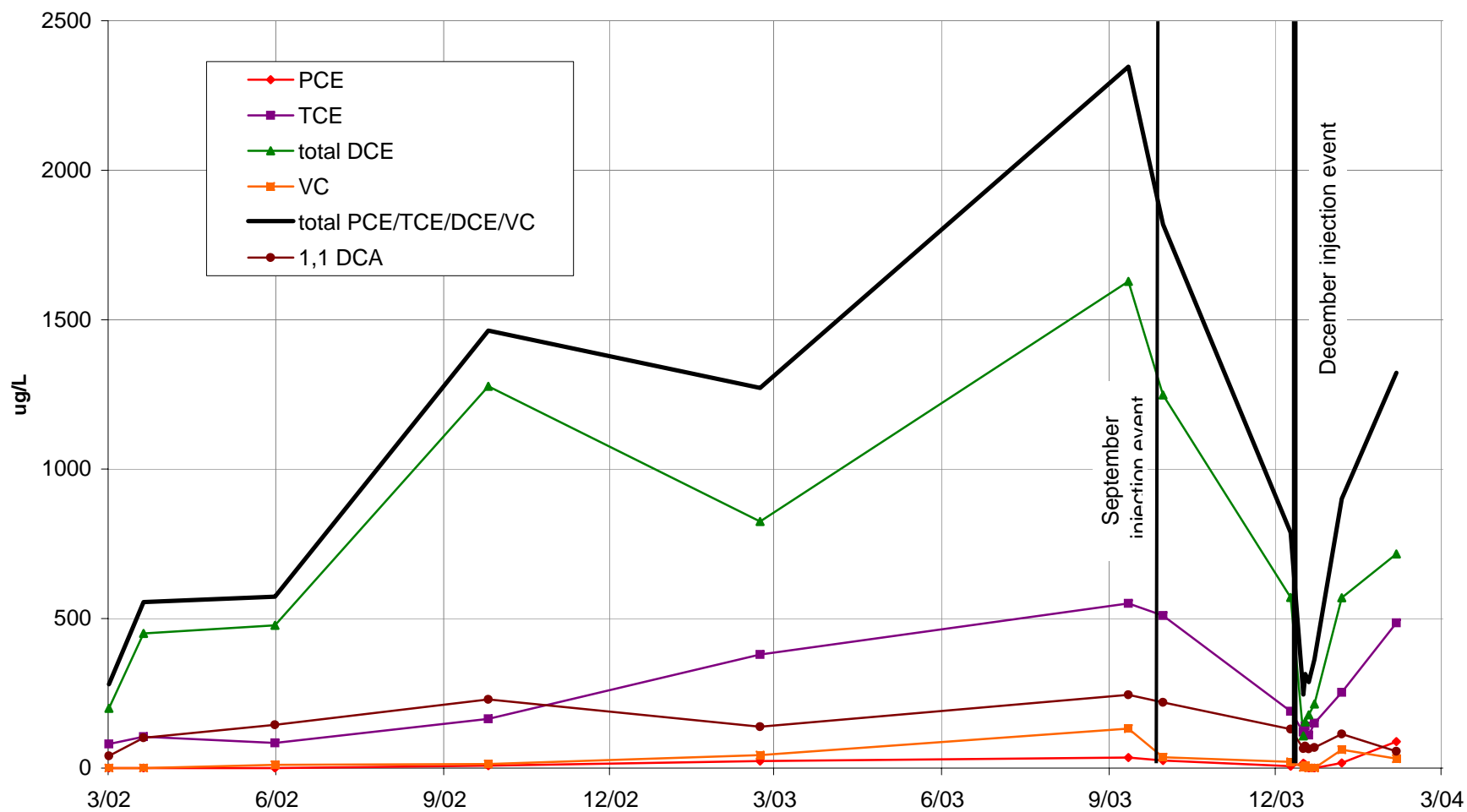


Figure 2. Concentrations of consistently detected VOCs in SB51L-02-3.

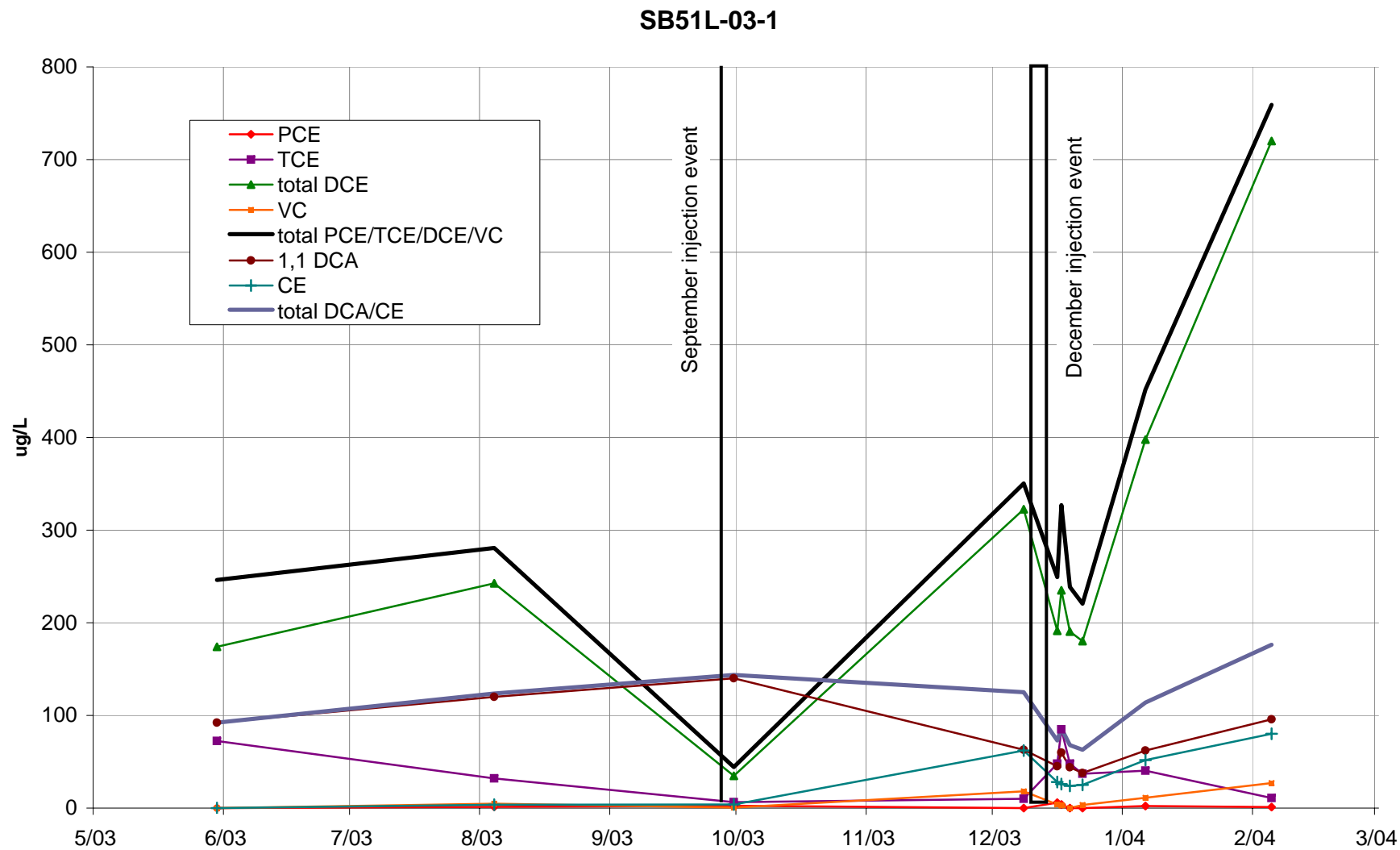


Figure 3. Concentrations of consistently detected VOCs in SB51L-03-1.

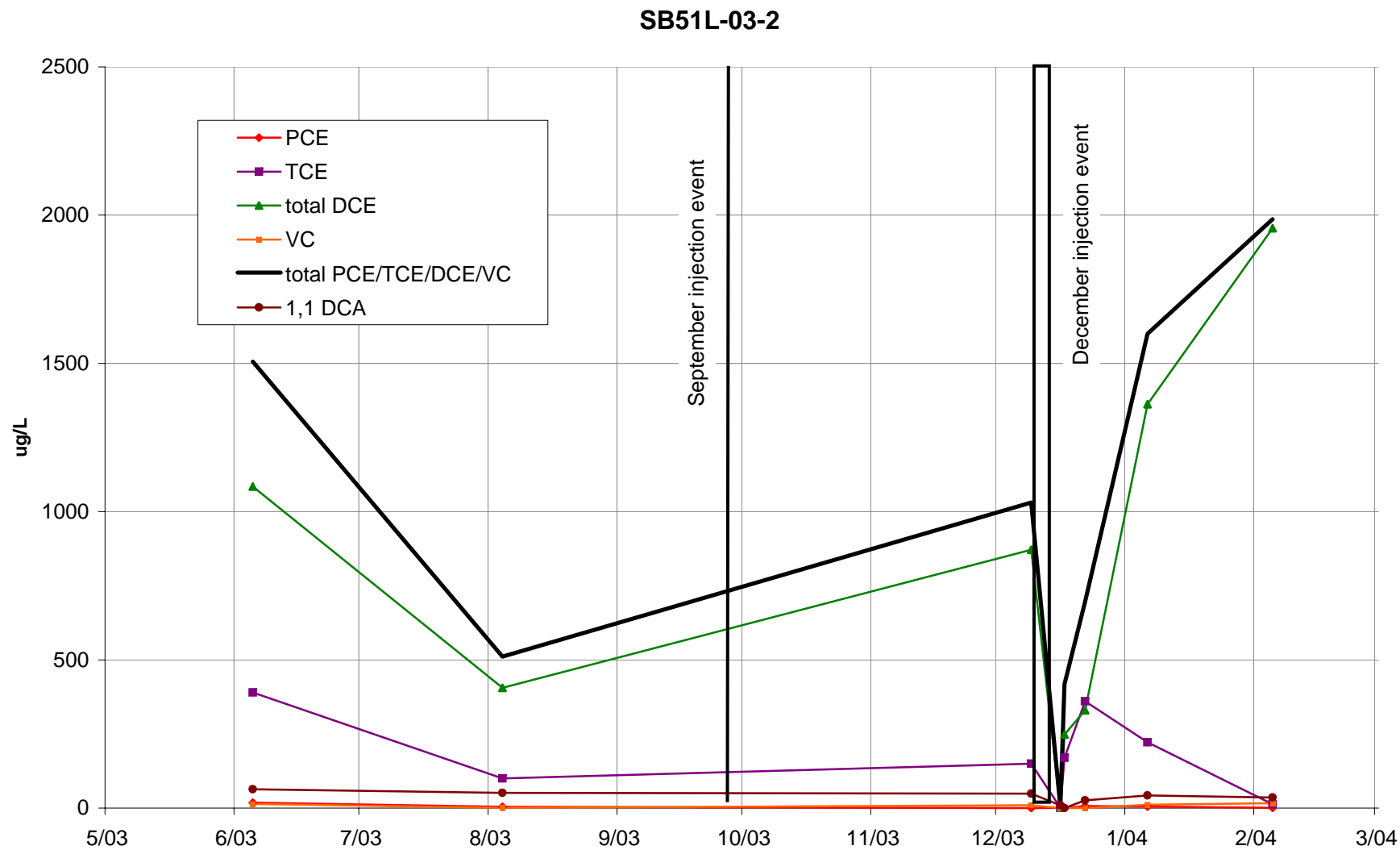


Figure 4. Concentrations of consistently detected VOCs in SB51L-03-2.

SB51L-02-3

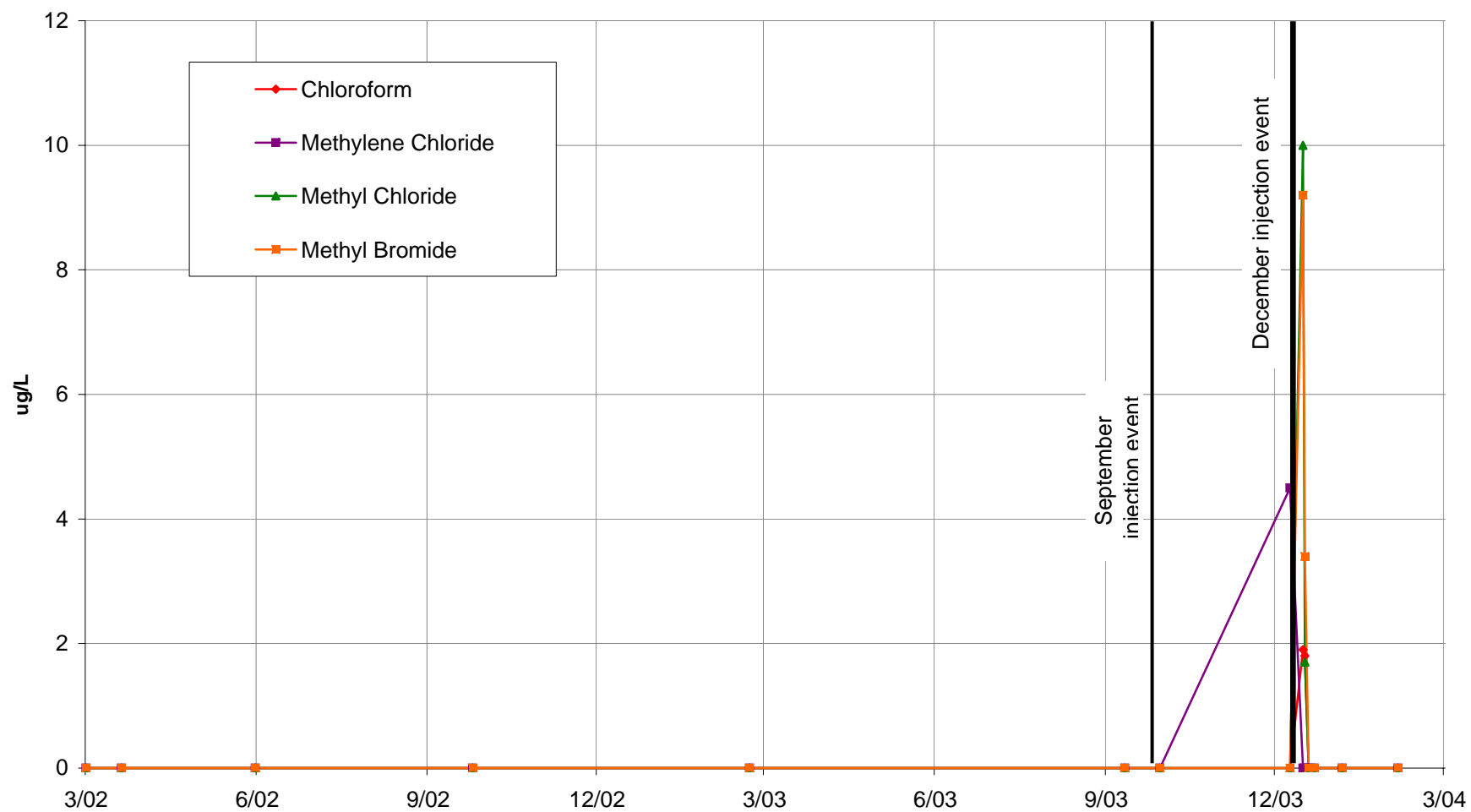


Figure 5. Concentrations of sporadically detected VOCs in SB51L-02-3.

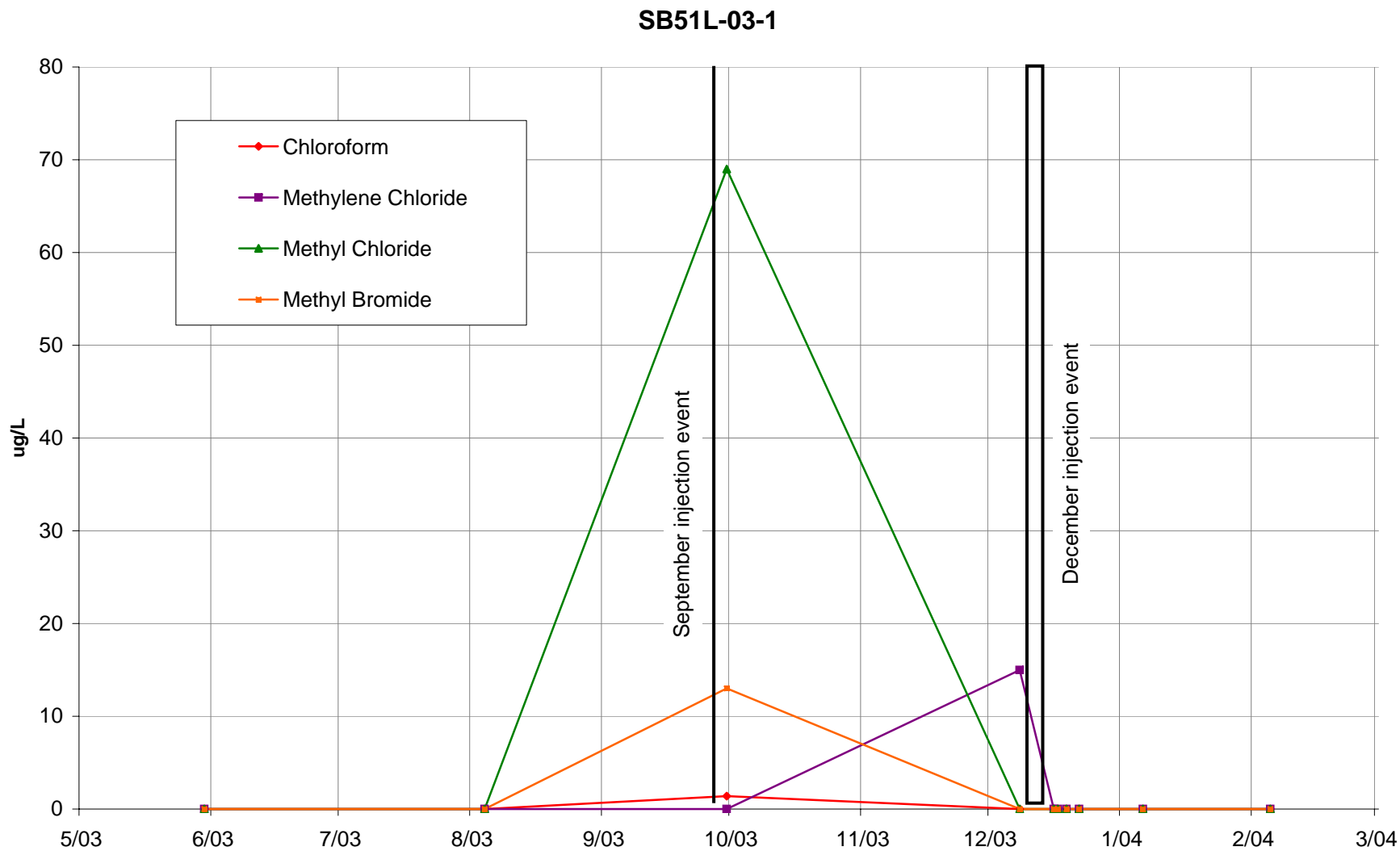


Figure 6. Concentrations of sporadically detected VOCs in SB51L-03-1.

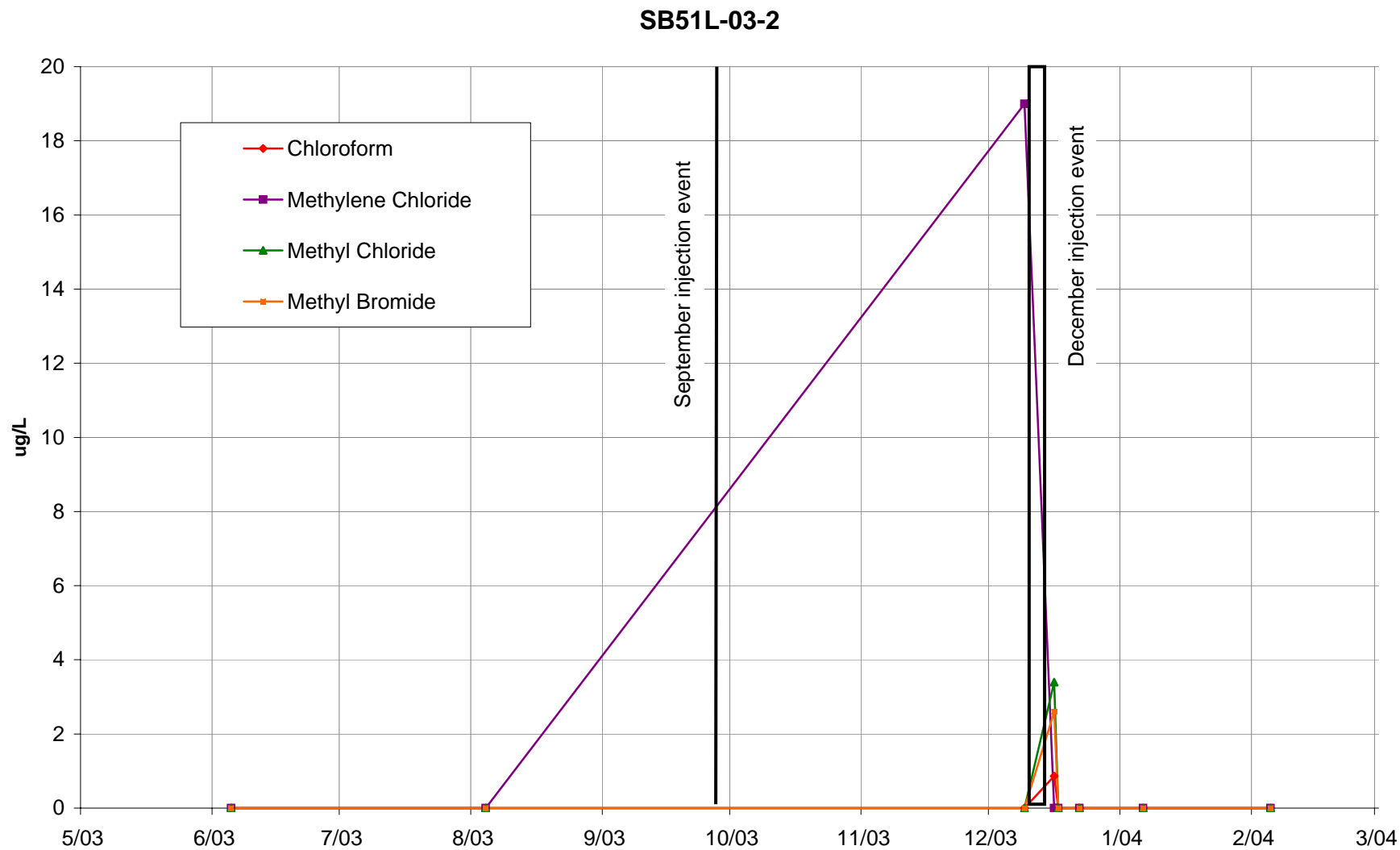


Figure 7. Concentrations of sporadically detected VOCs in SB51L-03-2.



Figure 8. Locations of cross section C-C' and new borings SB51L-04-1 and SB51L-04-2.

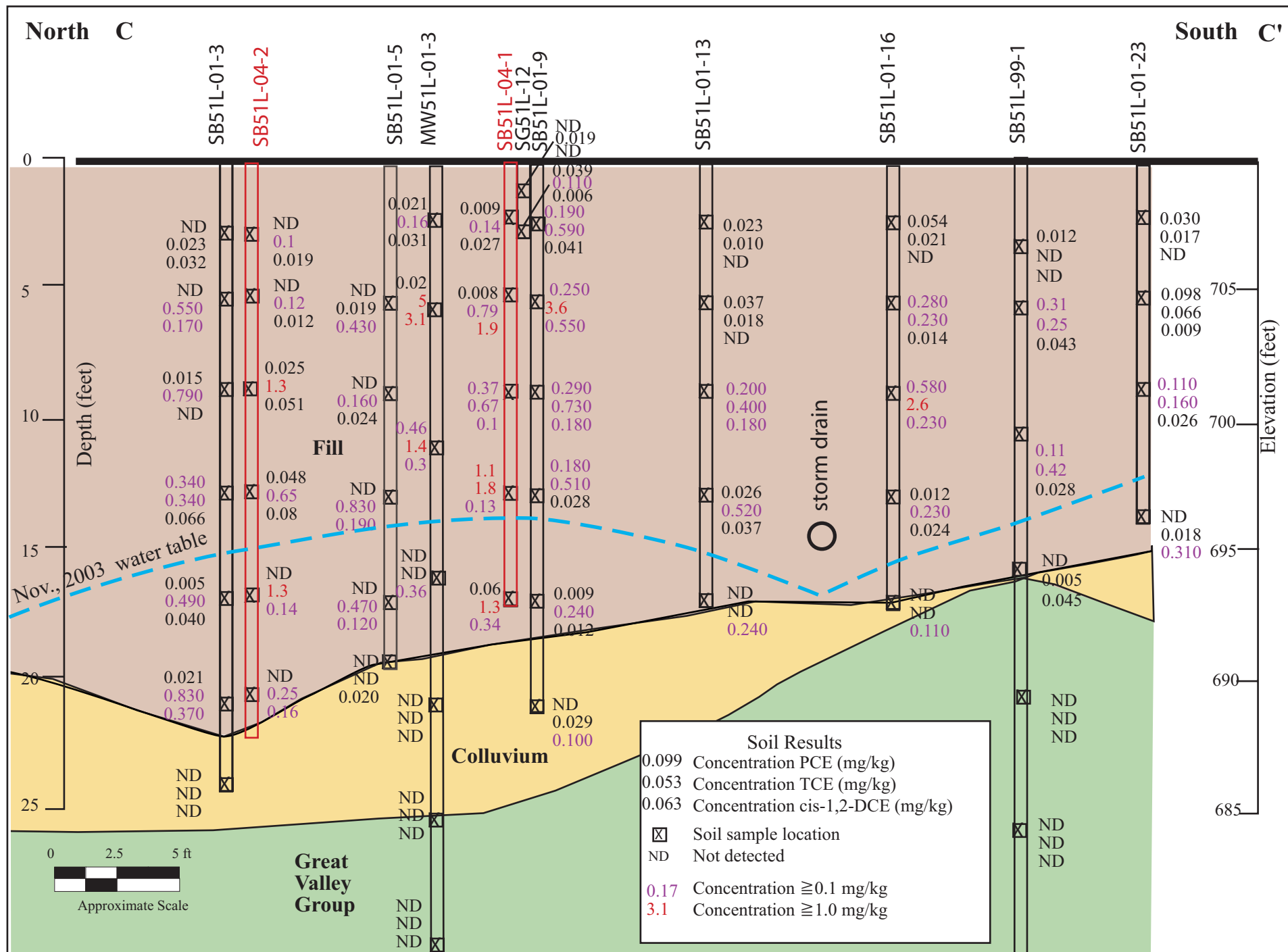


Figure 9. Cross section C-C', Building 51L area, showing concentrations of PCE, TCE and cis-1,2-DCE in soil (mg/kg).

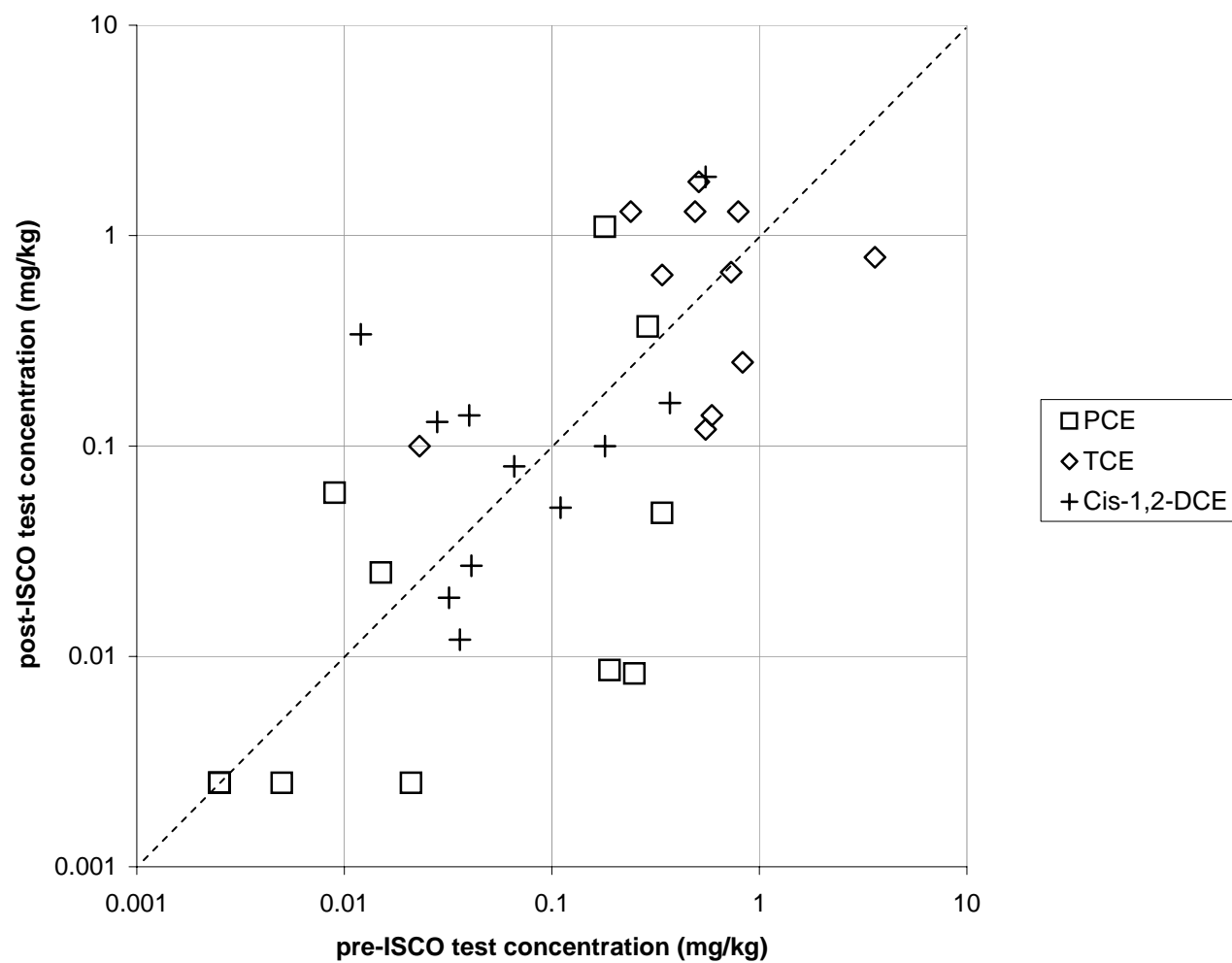


Figure 10. Comparison of the concentrations in soil of the most commonly detected VOCs prior to and after the ISCO test.

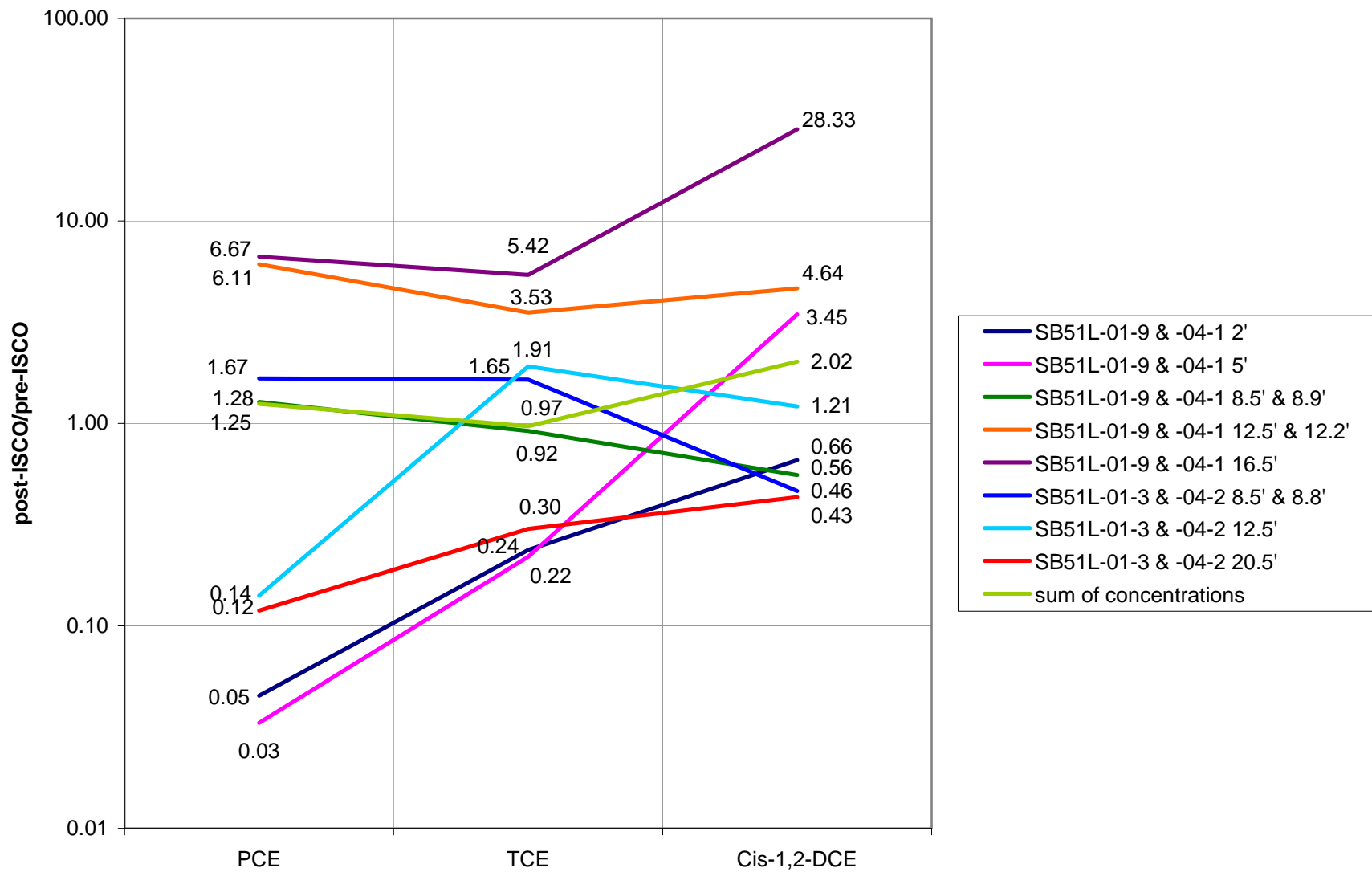


Figure 11. Comparison of PCE, TCE, and Cis 1,2-DCE concentration ratios for each pre- and post-ISCO test soil sample pair. Sample sets with nondetects are not shown.